

# METALLURGIA

THE BRITISH JOURNAL OF METALS

Vol. 62 No. 371

SEPTEMBER, 1960

Monthly: Two Shillings and Sixpence

▼ Steel Casting Ladle as supplied to The Steel Company of Wales

## STEELWORKS PLANT?

### REMEMBER THE SCOPE OF NEWTON CHAMBERS

Newton Chambers are fully equipped and have many years' experience in the design, manufacture and installation of a wide range of steelworks plant and ancillary equipment. May we discuss your requirements, particularly for the following?

#### STEELWORKS PLANT

- ☐ Steel Casting Ladles and Carriages up to 300 Tons Capacity
  - ☐ Hot Metal Ladles and Carriages
- ☐ Torpedo Type Hot Metal Ladles and Cars ☐ Slag Ladles and Carriages
- ☐ Ingot Casting Cars ☐ Ingot Transfer Cars ☐ Slab Transfer Bogies
- ☐ Coil Carrying Bogies ☐ Scrap Bogies ☐ Charging Boxes and Bogies
- ☐ Roll Transfer Bogies ☐ Special Transport Equipment
- ☐ Gas Cleaning Plant (Electrostatic or Venturi Type) ☐ Recuperators

#### BLAST FURNACE PLANT

- ☐ Blast Furnace Plant ☐ Hot Blast Stoves
- ☐ Slag Ladles and Carriages
- ☐ Pig Casting Machines ☐ Gas Cleaning Plant ☐ Gasholders



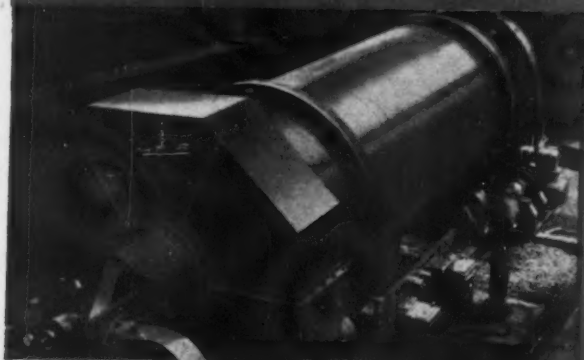
NEWTON CHAMBERS & CO LTD · ENGINEERING DIVISION · THORNCLIFFE · SHEFFIELD

# STORDY - HAUCK

## SELF-PROPORTIONING OIL BURNERS

Stordy-Hauck self-proportioning oil burners are designed for better oil combustion, accurate control of furnace temperature and atmosphere and maximum fuel economy.

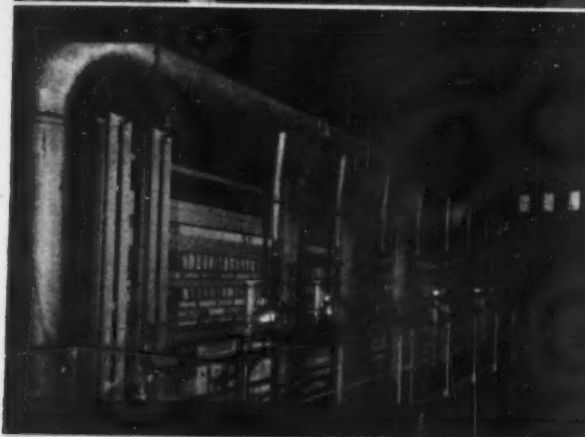
Brochures are available to illustrate the full range of Stordy-Hauck burners.



### ASPHALT PLANT

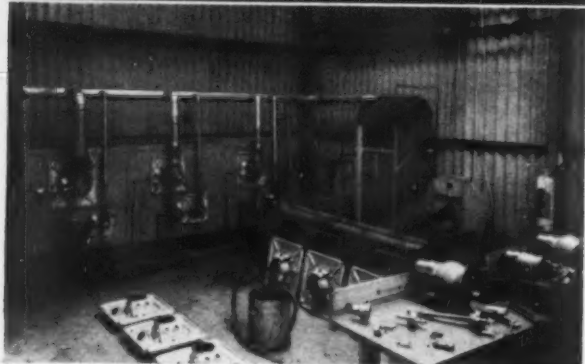
A Stordy-Hauck self-proportioning oil burner applied to a rotary dryer and asphalt plant.

This burner more than meets the operating temperature range required for varying materials handled.



### GLASS MELTING

Stordy-Hauck self-proportioning oil burners are fitted to this Hartford Empire Unit glass melting installation, comprising 18 burners all under full automatic control.



### TESTING

Stordy manufacture at their Wombourn Works a full range of Hauck fully self-proportioning oil burners with full facilities for experimental work and for full testing of production burners before despatch.

**STORDY ENGINEERING LIMITED**

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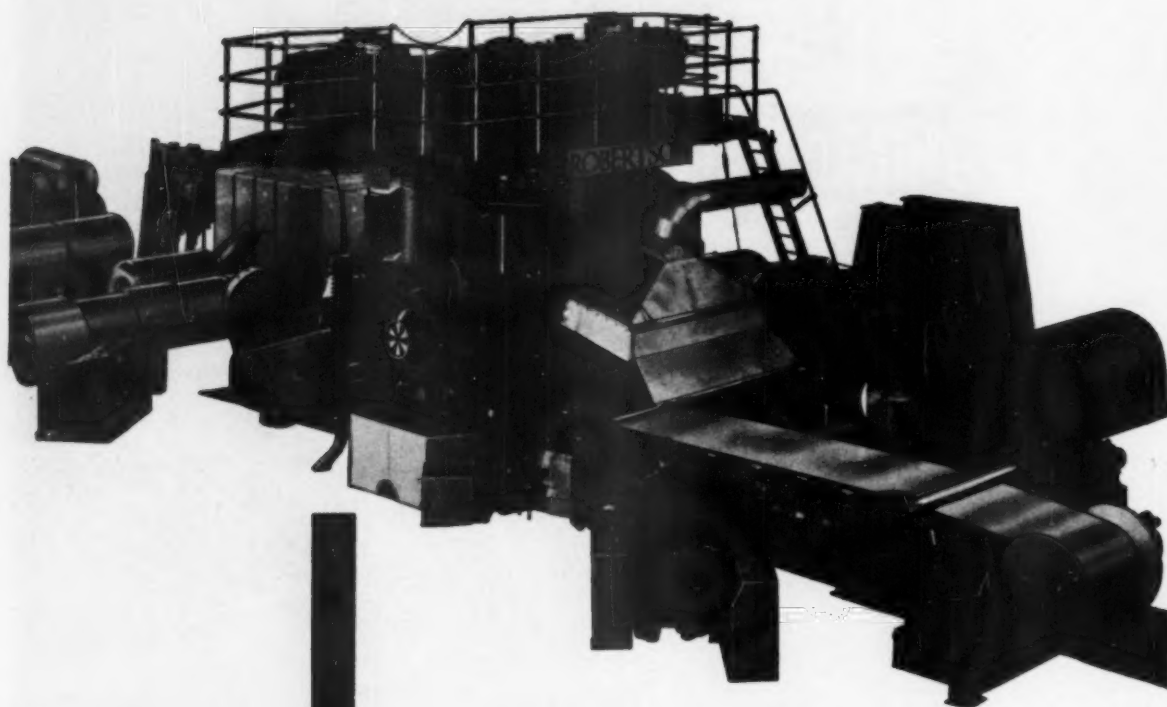
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W.S. 101

# ROBERTSON

## in BELGIUM



**Four-high Reversing Cold-Rolling Mill  
for aluminium and light alloy strip up to  
50 in. finished width.**

*Photograph by courtesy of Société Industrielle de  
l'Aluminium, Duffel, Belgium.*

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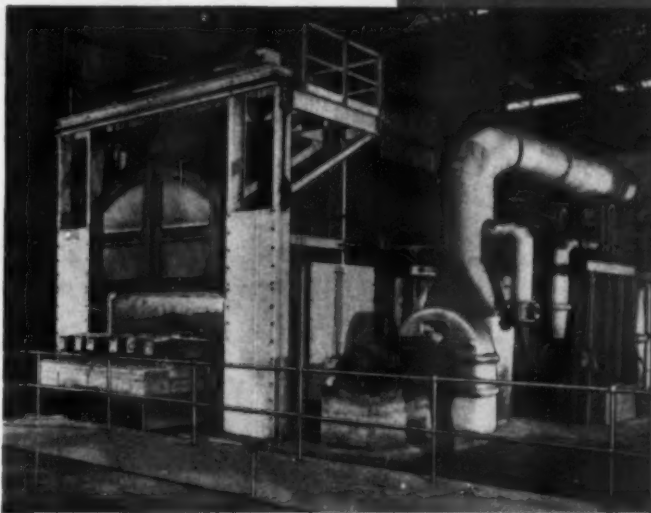
# PRIEST

## CONTINUOUS BOGIE TYPE REHEATING FURNACE

Illustrated is a Gas-fired Furnace supplied to Jessop-Saville Ltd. Brightside Works, Sheffield, for reheating alloy steel ingots prior to rolling.

*We also specialise in the design and construction of:—*

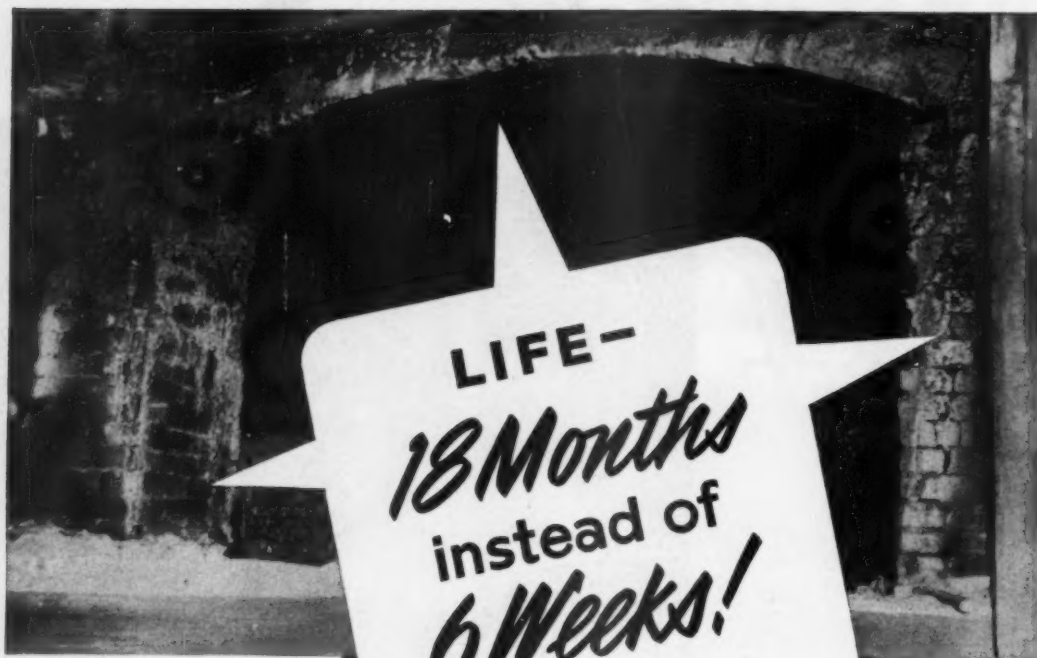
Open Hearth Furnaces  
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Continuous Multi-zone Bloom and Slab Re-heating Furnaces  
Continuous Bogie type Ingot and Slab Heating Furnaces  
Furnaces for Aluminium Melting, Coil Annealing and Slab Re-heating  
Forge and Heat Treatment Furnaces  
Stress Relieving Furnaces  
Shipyard Plate and Bar Furnaces



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also at KELHAM ISLAND WORKS • SHEFFIELD 3

# *Sweeping Economies* follow use of **GR SILLMAX '54'**



GR Technical Service  
Department is available for  
consultation by users in the  
selection and application of  
these and other refractories.



**Everything in  
Furnace Linings**

The illustration shows the condition of the pillars and arches of a forge furnace in France after 18 months service. The refractories used were GR Sillmax '54' bricks and they replaced 40%  $Al_2O_3$  firebricks which gave an average life of 6 weeks.

GR Sillmax '54' are characterised by low porosity high spalling resistance, extremely high mechanical strength and toughness with a marked resistance to abrasion. Recommended for reheating furnaces, Soaking pit walls and roofs, Electric furnace roofs, Regenerator chambers.

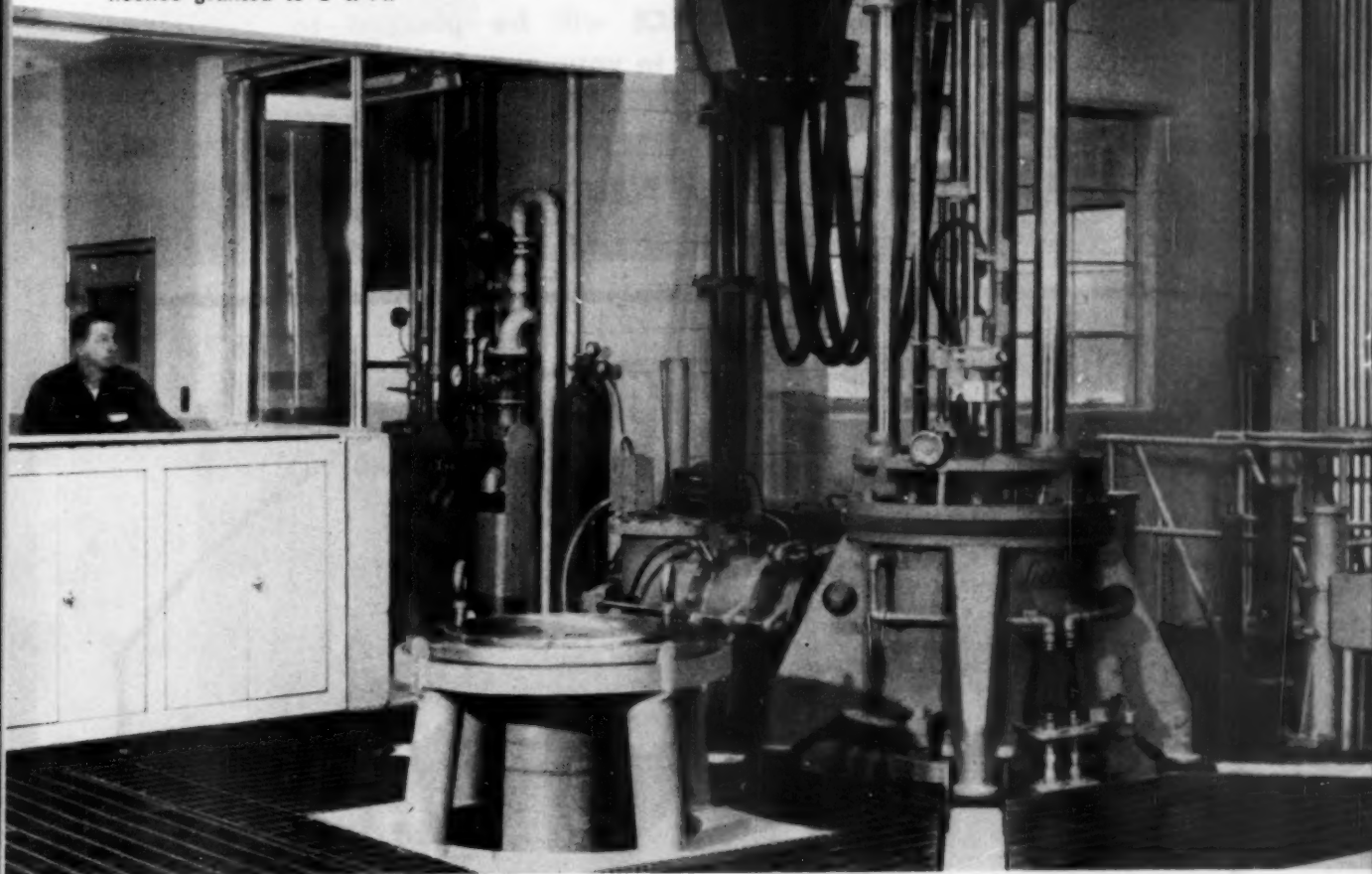
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*Lectromelt*



**Stein & Atkinson Ltd.**

**Westminster House Kew Road Richmond Surrey**

A 20" x 8500 pound furnace installed by Lectromelt at Latrobe Steel Co., Latrobe, Pennsylvania.

**FURNACE ENGINEERS**

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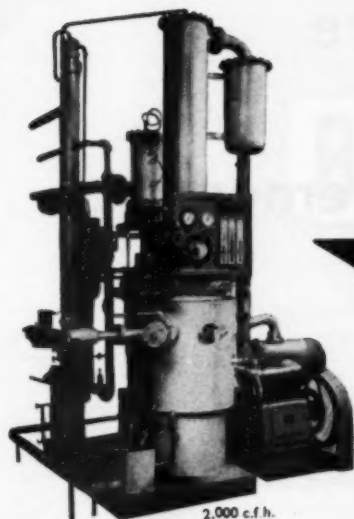


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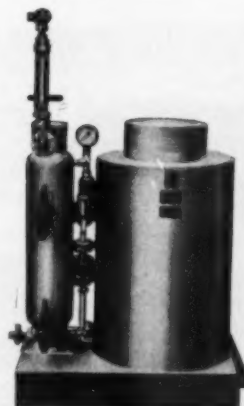


### **Gibbons Mahler-v-Linde**

Atmosphere generators with a wide range of applications in many industries. In the metal industry for bright annealing, brazing, sintering, gas carburising, bright hardening, etc.

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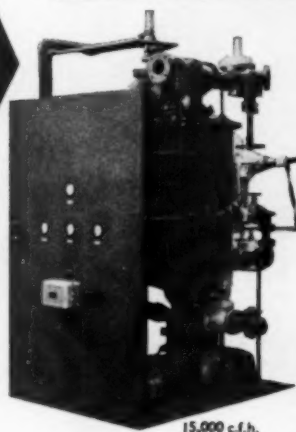
For bright treatment of stainless acid resisting and heat resisting steels, and for brazing and sintering generally.



150 c.f.h.

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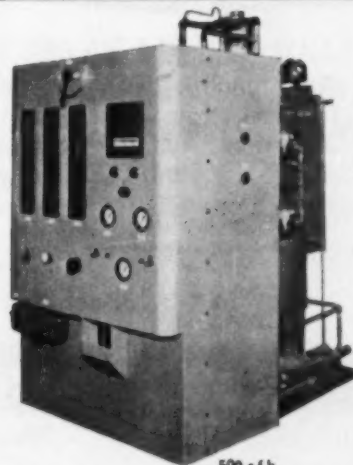
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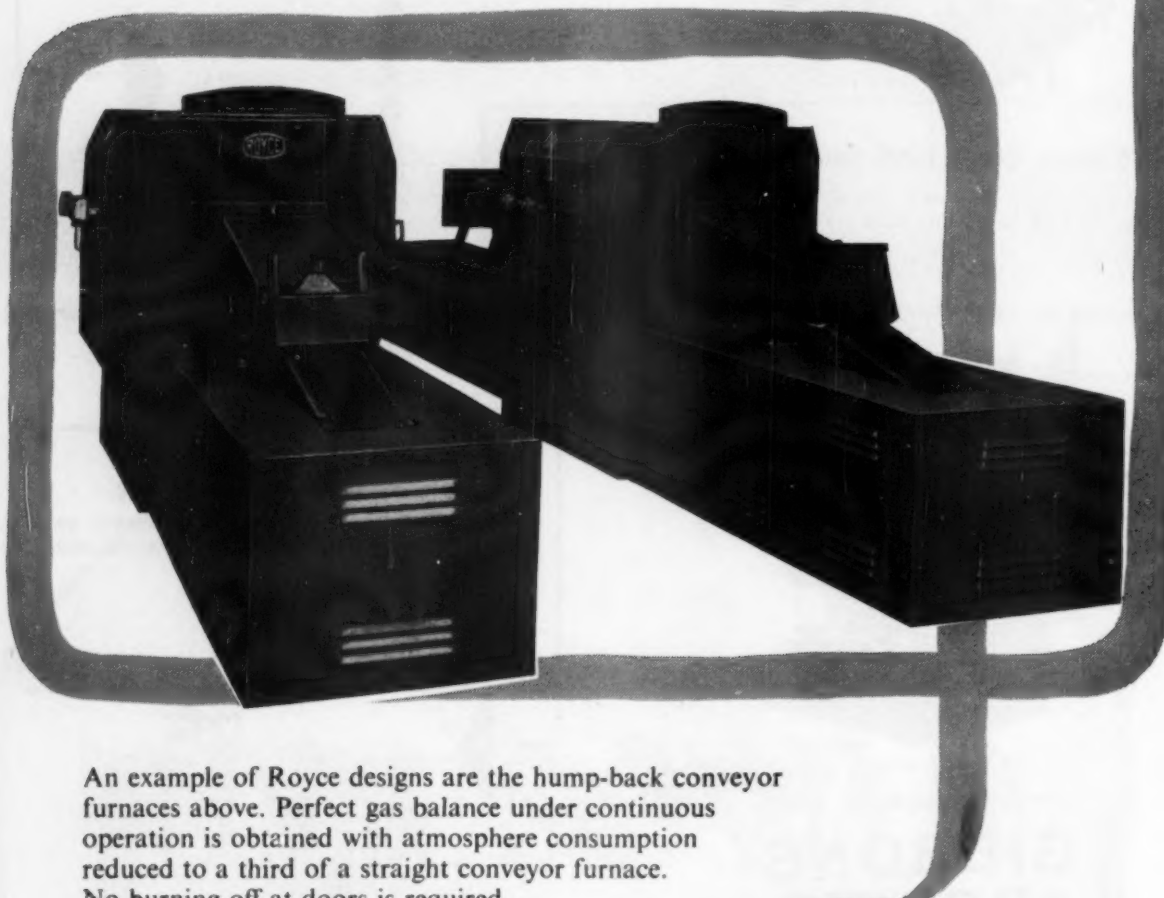
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An example of Royce designs are the hump-back conveyor furnaces above. Perfect gas balance under continuous operation is obtained with atmosphere consumption reduced to a third of a straight conveyor furnace. No burning off at doors is required.

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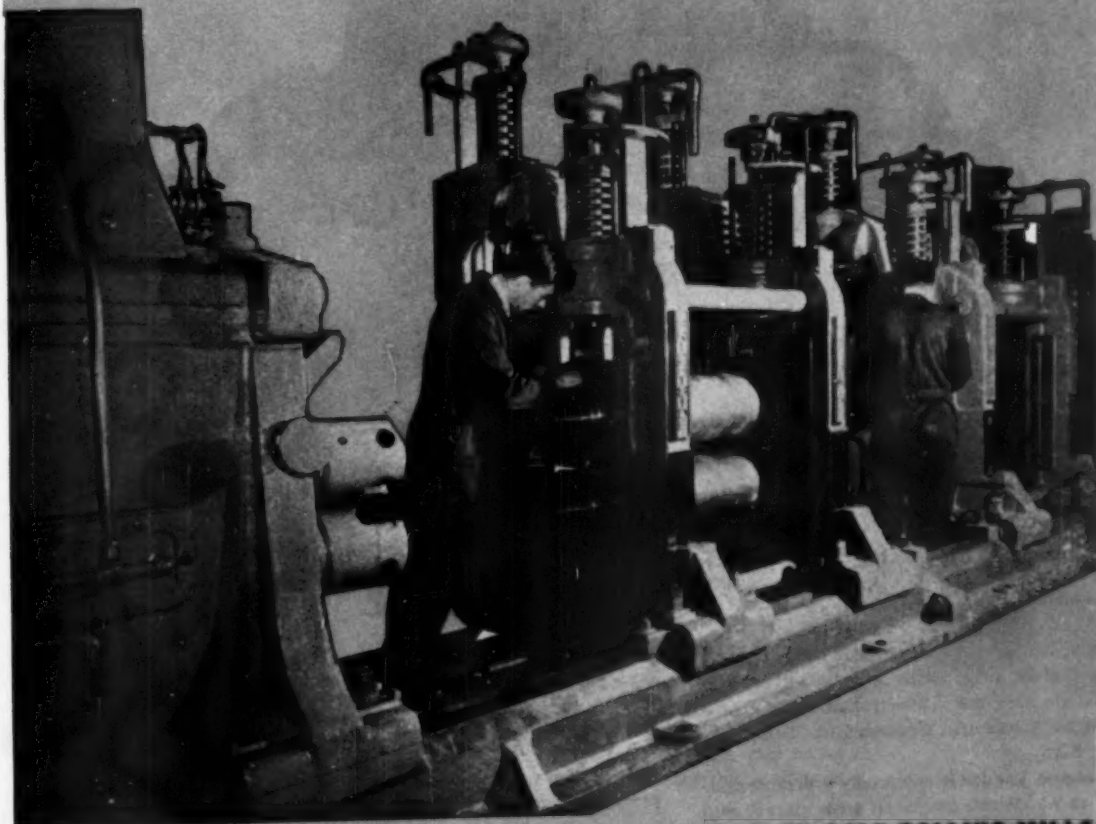
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# NICKEL ALLOY STEELS

ensure reliability in loading



Samson Loader Model MC3



Bevel pinion and crown wheel assembly for gathering head.

Economical coal production depends upon rapidly removing coal or stone when it has been brought down. To do this the Samson Loader has been developed by Mavor and Coulson Ltd., Bridgeton, Glasgow, S.E.

The Samson Loader is essentially a flexible conveyor on to which two gathering arms quickly pull loose material. The delivery end of the jib can be varied from 2 ft 1 in to 8 ft 4 in high, and peak capacity working for Model MC3 is up to 5 tons of coal per minute.

To achieve reliability under the difficult conditions in coal mines, the designers specified nickel alloy steels notably En25, En36B and En24 or equivalents for high impact values. These steels are used for gear shafts and other components.

## TYPICAL MECHANICAL PROPERTIES OF

# EN25

These illustrate its strength and toughness in massive sections

SIZE	HEAT TREATMENT	YIELD STRESS L.s.i.	MAXIMUM STRESS L.s.i.	ELONGATION per cent	1200 ft. lb.
2½ in. dia.	Oil quenched 830°C. tempered 590°C.	75	78	17	44
2½ in. dia.	Oil quenched 830°C. tempered 650°C.	58	63	21	68
4 in. dia.	Oil quenched 830°C. tempered 650°C.	56	61	21	69
6 in. dia.	Oil quenched 830°C. tempered 650°C.	55	61	22	63

By utilising the better properties obtainable in nickel alloy steels, dimensions can be reduced, lighter constructions produced, distortion through heat-treatment minimised and reliability and economy achieved.

Please send for our publications entitled, 'The Mechanical Properties of Nickel Alloy Steels' and 'The Case Hardening of Nickel Alloy Steels'.

## MOND NICKEL



THE MOND NICKEL COMPANY LIMITED, THAMES HOUSE, MILLBANK, LONDON, SW1

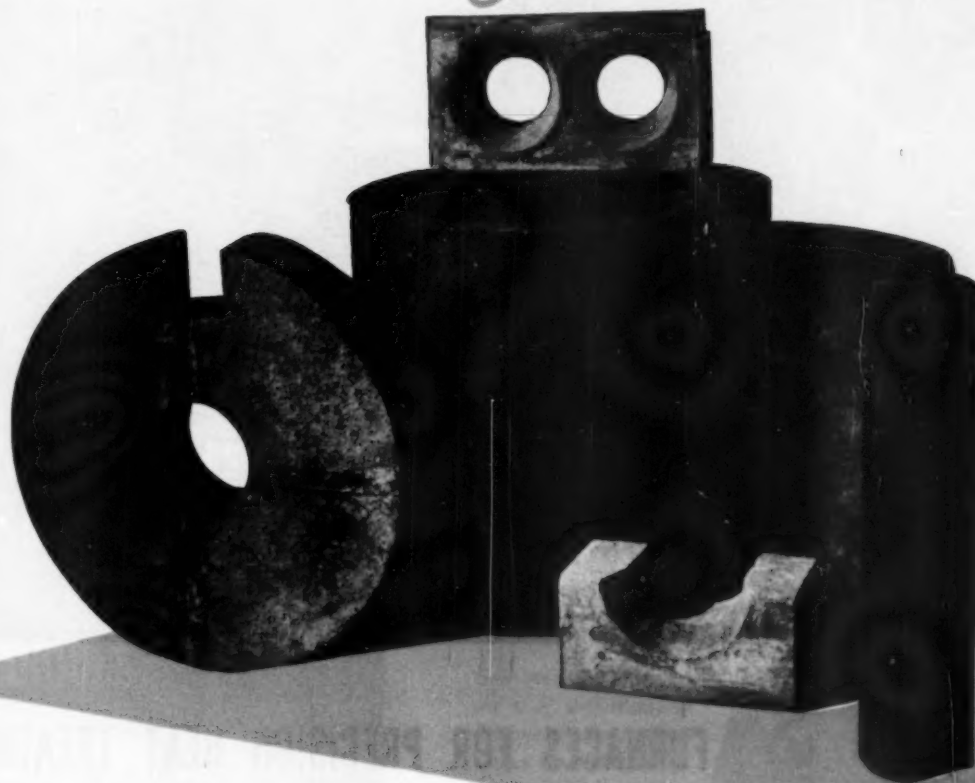


TGA 21/2/10



# STEIN

## Refractories



### REFRACTORY CONCRETE

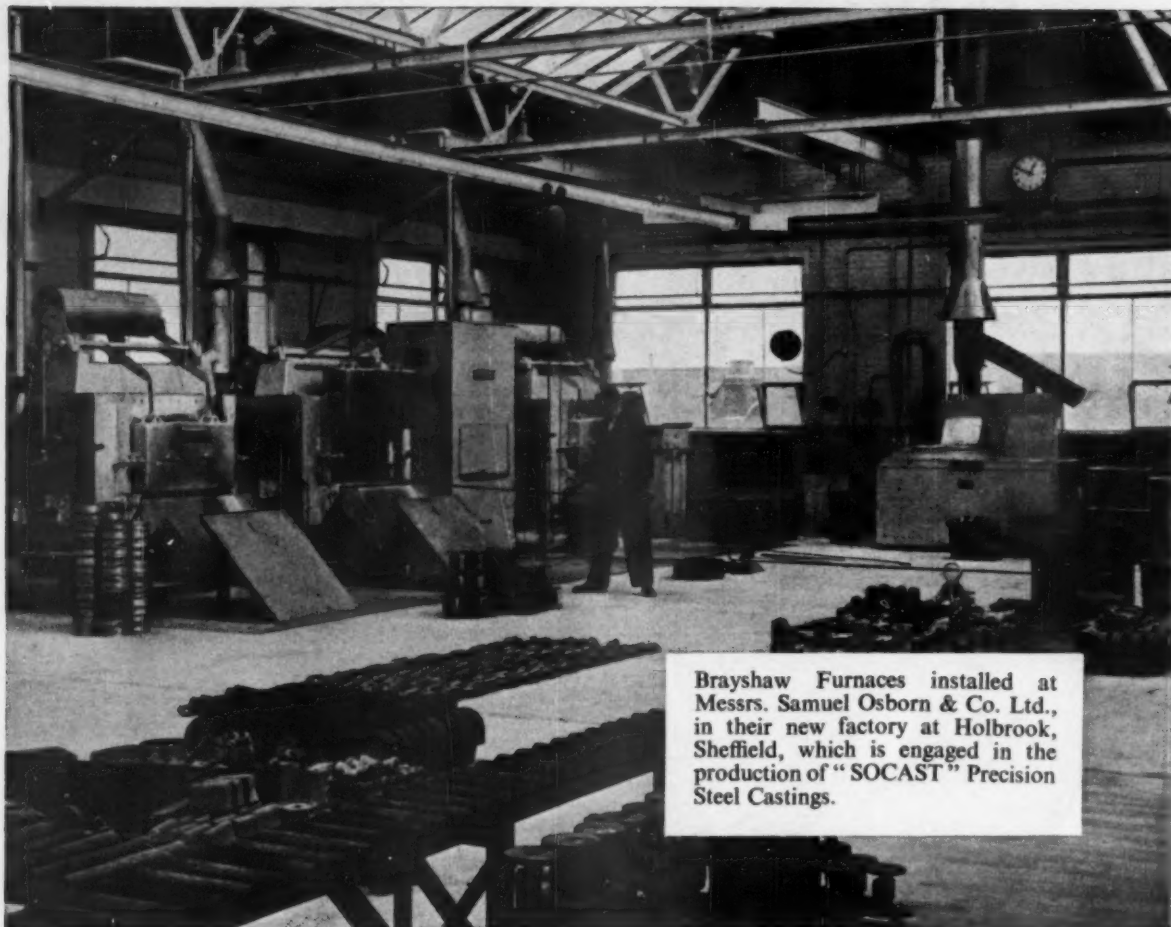
Refractory Castables are poured and cast like structural Concrete, and being readily available are finding many applications, particularly for preparation of special shapes at site.

	HOW SUPPLIED	REFRACTORINESS	TYPE OF SET	THERMAL HARDENING TEMPERATURE	MAXIMUM TEMPERATURE OF USE	Lb./Cu. Ft.
Stein Refractory Concrete						
No. 13	Dry	1420°C	Hydraulic	1200°C	1350°C	120
No. 14	Dry	+ 1500°C	Hydraulic	1200°C	1450°C	120
No. 16	Dry	+ 1750°C	Hydraulic	1200°C	1600°C	160
No. 17	Dry	+ 1750°C	Hydraulic	1200°C	1700°C	160
No. 18	Dry	+ 1750°C	Hydraulic	1200°C	1800°C	160
Stein Chrome Concrete	Dry	+ 1750°C	Hydraulic	1200°C	1500°C	180

*Use our advisory service based on 70 years experience in the refractory field—it can improve your furnace efficiency. For further information write, phone or call:*

**JOHN G. STEIN & CO. LTD. Bonnybridge, Scotland**

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Brayshaw Furnaces installed at Messrs. Samuel Osborn & Co. Ltd., in their new factory at Holbrook, Sheffield, which is engaged in the production of "SOCAST" Precision Steel Castings.

# Brayshaw

## FURNACES FOR PRECISION HEAT TREATMENT

Once again furnace design by Brayshaw is playing a vital part in a new industrial advance . . .

In the development of their "SOCAST" Precision Steel Casting techniques and for the increasing production flow from their new factory, Messrs. Samuel Osborn & Co. Ltd., have specified Brayshaw Furnaces.

The choice demonstrates a confidence in Brayshaw design and manufacturing ability that is shared in increasing measure by forward looking concerns the world over.

Expert consultation on new projects, re-equipment, conversion, repair and general maintenance of all types of plant is immediately available on request, please do not hesitate to . . .

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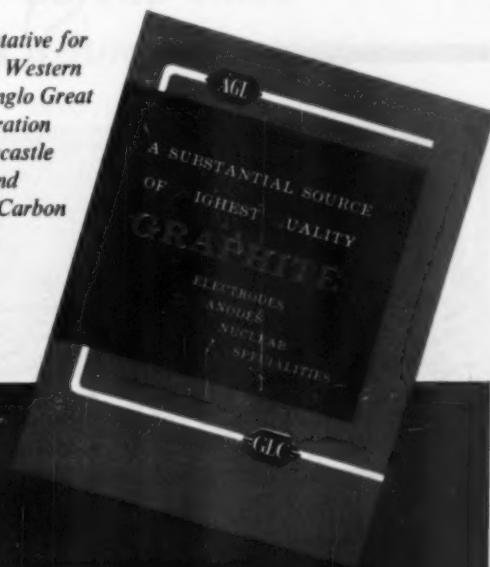
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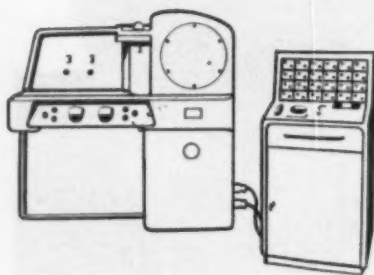
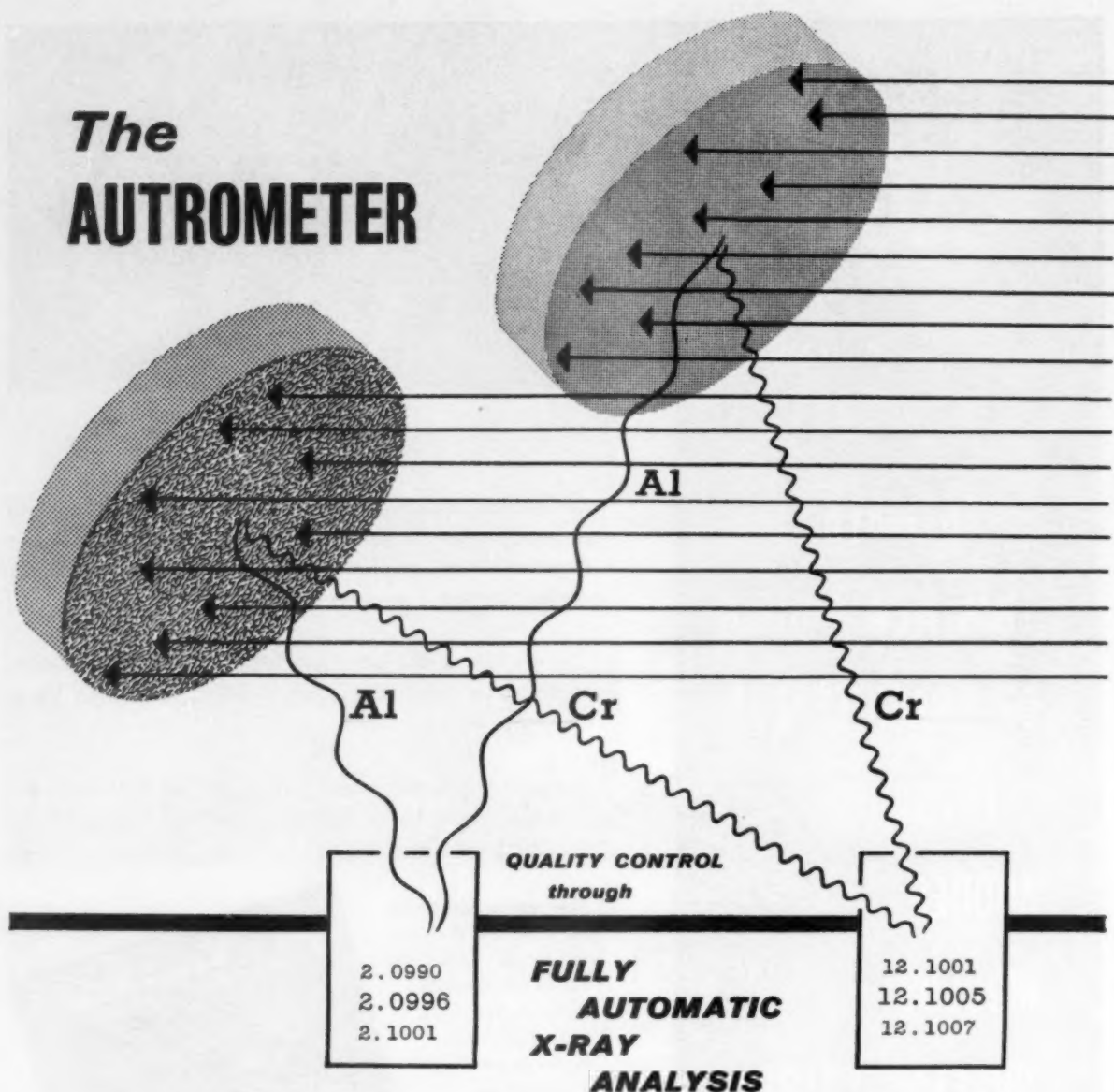


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Would metals with such characteristics make your production better? Wild-Barfield—NRC vacuum melting furnaces enable you to develop materials specially suited to your needs. Backed by the experience of the National Research Corporation, who have built and operated more high vacuum furnaces than any other company in the world, Wild-Barfield—NRC vacuum equipment covers most needs. Write for details of the range.

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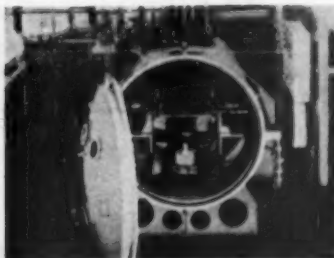


NRC is the trade-mark of the National Research Corporation, registered in the United States Patent Office.

## COULD A VACUUM FURNACE DO THE SAME FOR YOUR PRODUCTS?



Model 2355 Vacuum Induction Furnace with melting capacity of 50 pounds of steel. Other standard furnaces have capacities of 12 to 3,000 pounds.



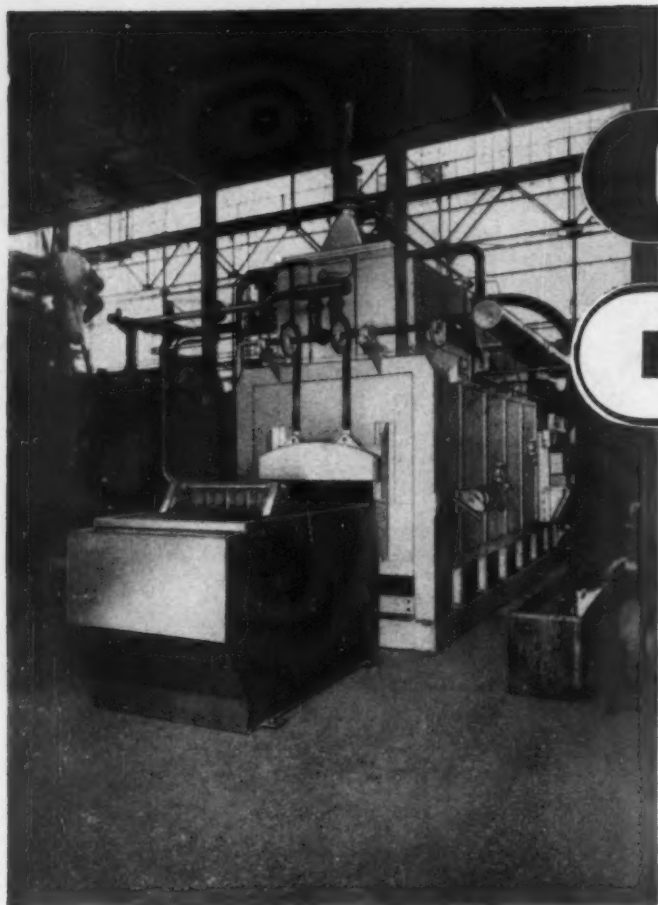
Model 2705 Non-Consumable Arc Skull Furnace with a capacity of 50 pounds of titanium. Other standard vacuum arc furnaces have capacities of 8 to 10,000 pounds of titanium.



**for all heat-treatment purposes**

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# GIBBONS

Patent Nos. 637,337. 646,691. 606,401.

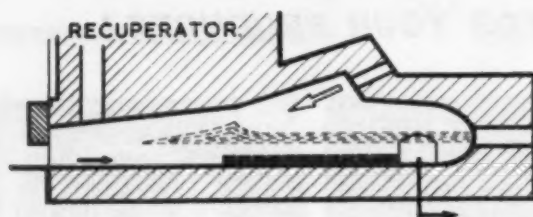
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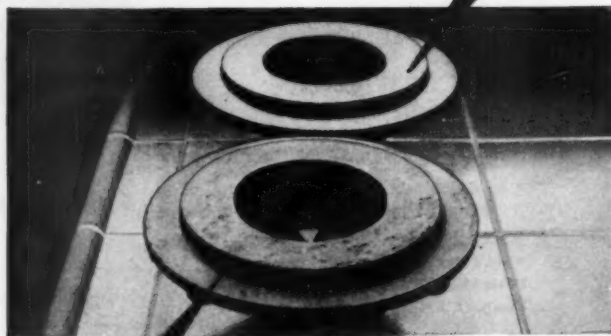
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**ref: refractories**



When it is a question of making a decision as to the best refractory bricks to use for a certain job, you need the best advice you can get.

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Our experience, both as manufacturers and users, is at your service at all times, and we invite you to write to us whenever you need information or advice.

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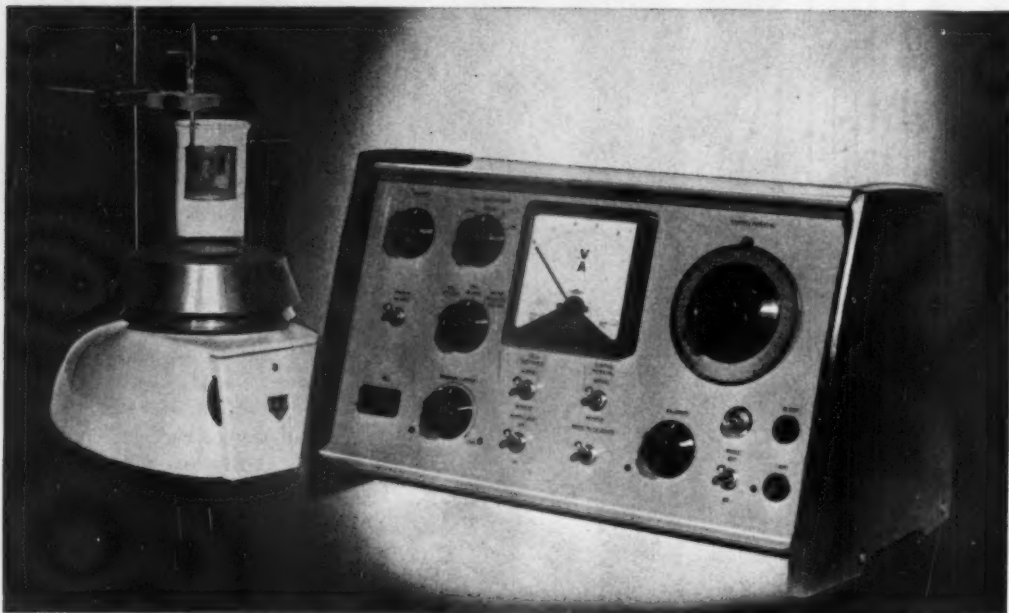
**A 1654**

## **Wadsworth Controlled Potential Electro-Depositor**

ELECTRO-DEPOSITION has long been recognised as an analytical and separative technique for solutions containing metallic ions. The method may be applied where the ionic concentrations of the metals present in solution vary widely, separation being best achieved where the potential of the working electrode relative to the solution is controlled, using a reference electrode as described

passing through the cell in such a sense that the potential error tends towards zero.

In order to ensure that thermal drift in the control amplifier is held at a very low level, a water-cooled heat sink is employed to maintain the transistors at a substantially constant temperature. The accuracy of control attained may be judged from the fact that the incremental error in electrode



independently by Sand & Fischer. Manual control of the electrode potential, although possible, is tedious and time consuming, with the result that automatic control circuits have been developed by a number of workers.

The Southern Analytical controlled potential electro-depositor is based upon a design due to N. J. Wadsworth of R.A.E., Farnborough, and is intended primarily for use with platinum electrodes. The circuit is fully transistorised in the interests of compactness and reliability and is capable of supplying a maximum controlled current of 10 amperes to the electrolysis cell. In operation, the required cathode potential is preset upon a built-in high grade potentiometer. Any error between the preset potential and that of a reference half-cell (e.g. saturated calomel), mounted immediately adjacent to the cathode, is applied to an amplifier which controls the current

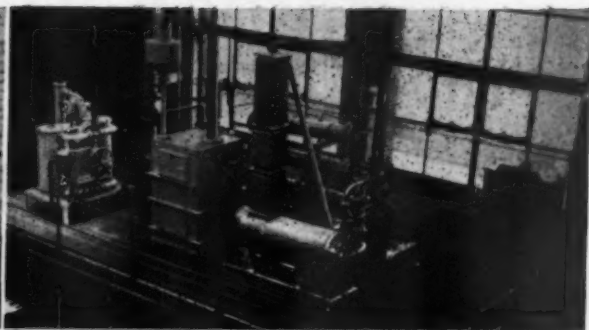
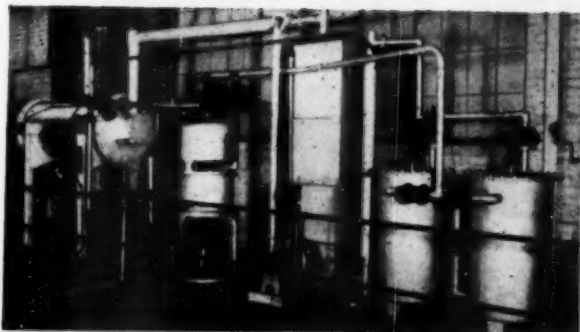
potential as a function of cell current has a mean value of 1.6 millivolts/ampere; thus, when the electrolysis current has fallen to the low levels obtaining towards the end of a deposition, extremely close control is established. It is noteworthy that the potential defined by the built-in potentiometer cannot be exceeded as cell current cuts off at zero potential error.

The apparatus is supplied complete with leads for connection to the user's own electrode assembly. If desired however, suitable standard accessory equipment can be provided.



SOUTHERN ANALYTICAL LIMITED CAMBERLEY SURREY TELEPHONE: CAMBERLEY 3401





## *Continuous* **BRIGHT** *annealing*

Whether the products be pressings, tubes or wire—ferrous or non-ferrous—Metaelectric continuous bright annealing furnaces bring a new and easier approach to manufacture.



Metaelectric bright annealing furnaces have all the latest features, designed for reliable operation — accurate temperature control, removable sections for easy inspection and maintenance, exceptional fuel economy. All these are backed by the facility for using the best form of atmosphere for the product. Town gas, propane and butane, kerosene or ammonia may be used as the fuel source for the atmosphere.

Top left : a conventional exothermic plant, with primary and reactivated secondary drying units and equipment for removal of sulphur compounds.

Top right : a fully automatic plant for producing commercially pure nitrogen with extremely low dewpoint.

Send for leaflet M.6A.

### **METALECTRIC FURNACES LTD.**

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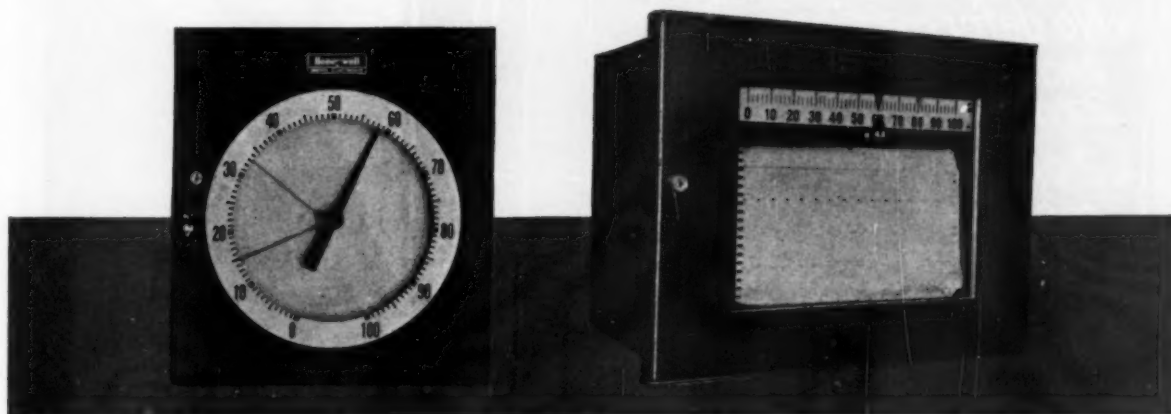
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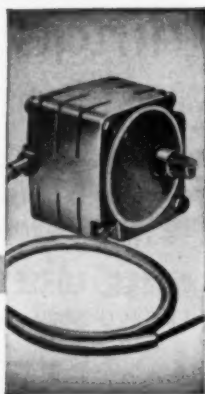
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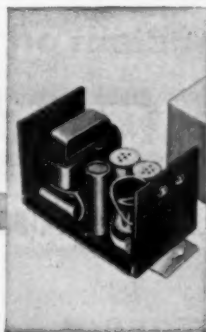


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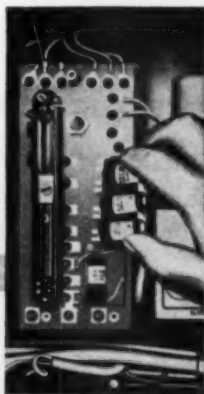
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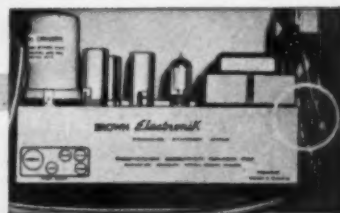
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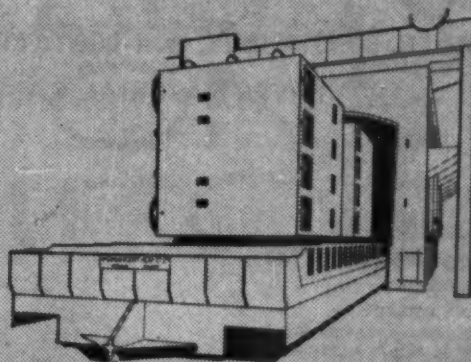
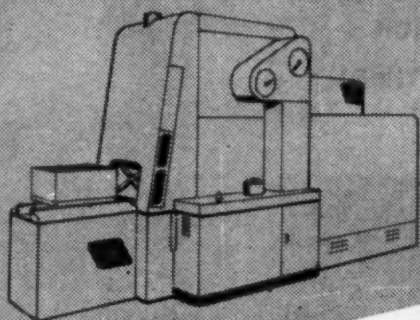
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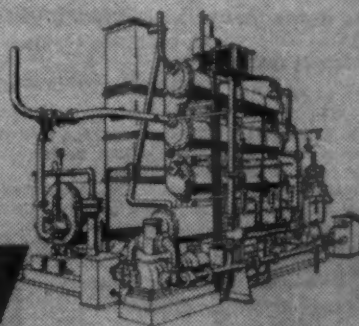
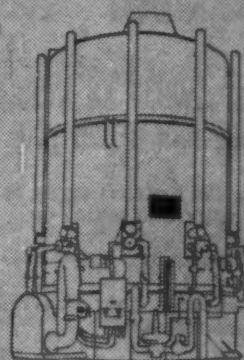
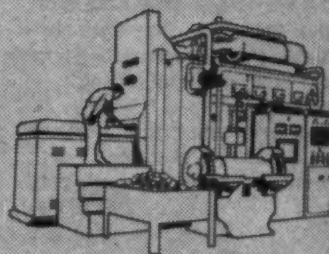
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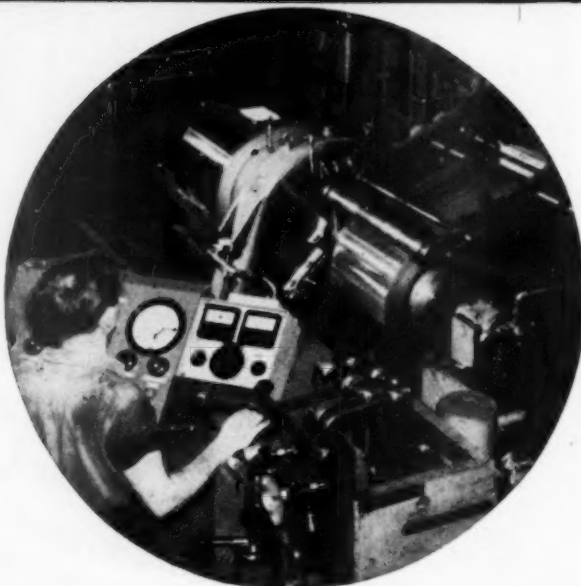
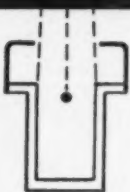
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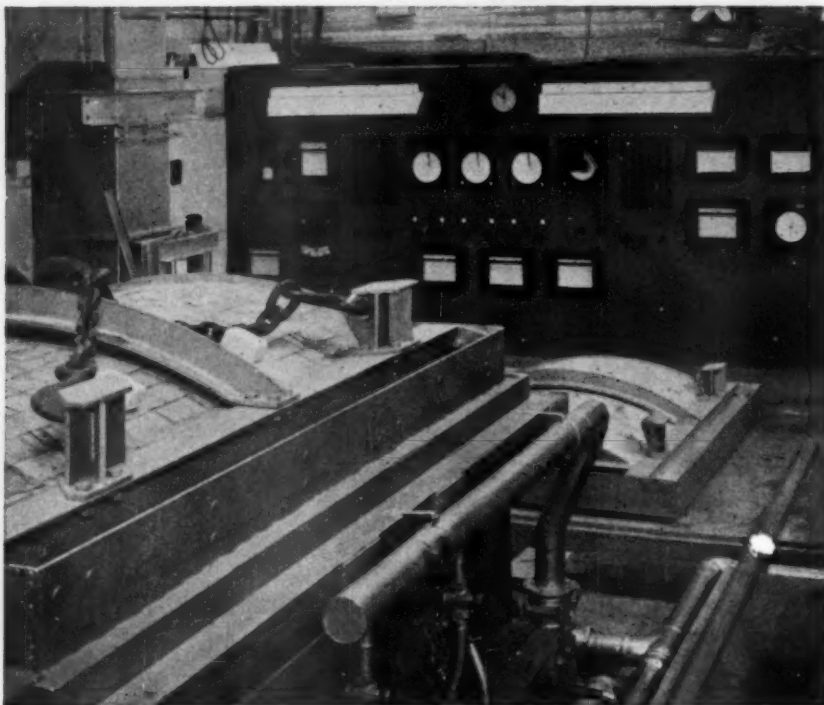
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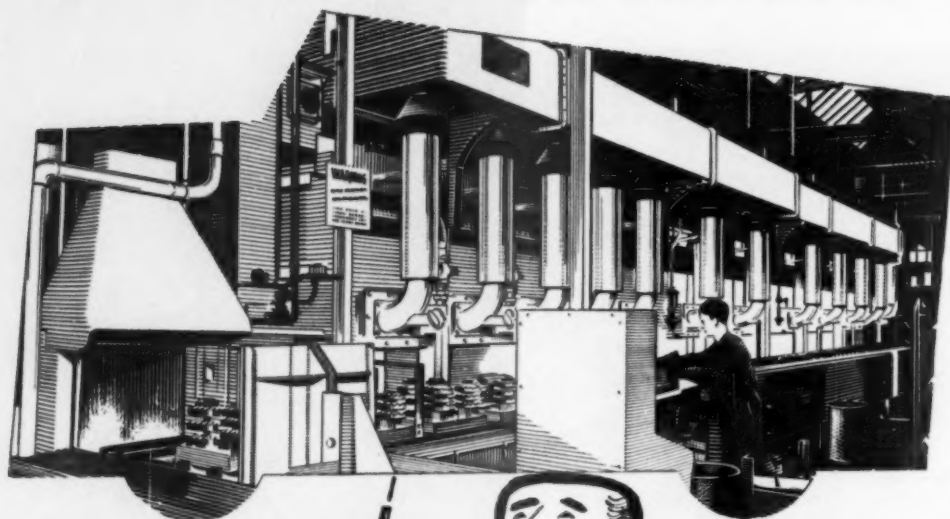
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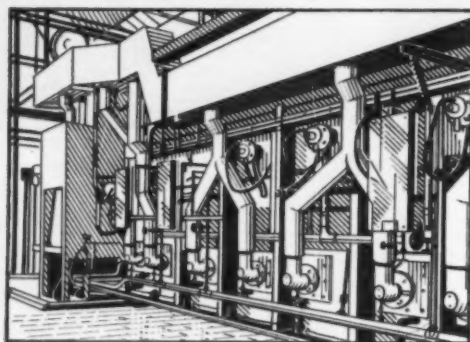
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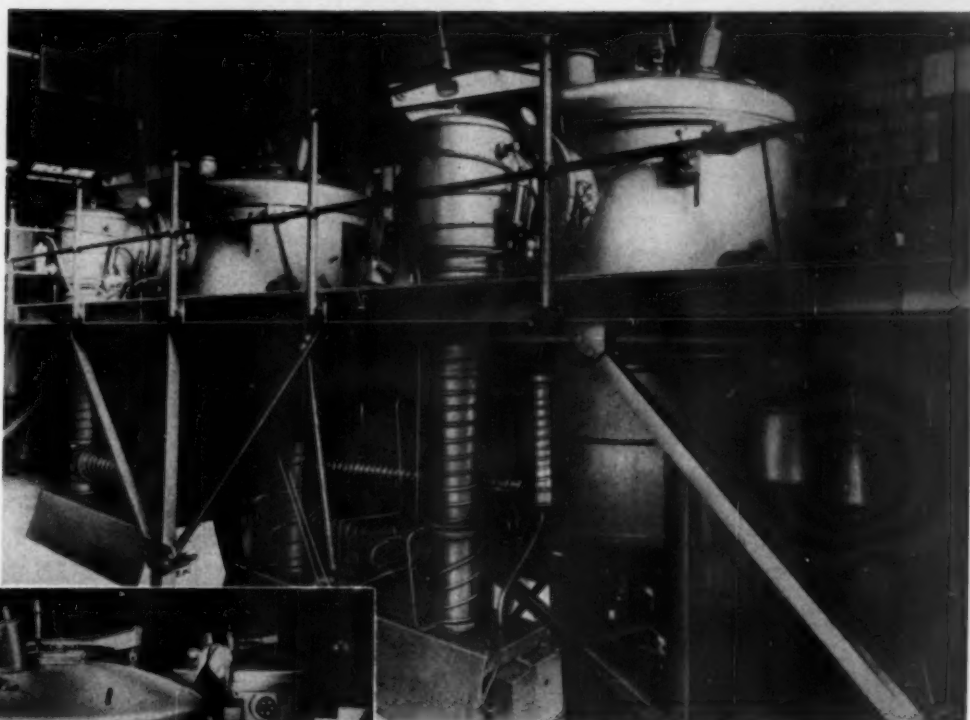
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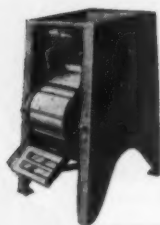
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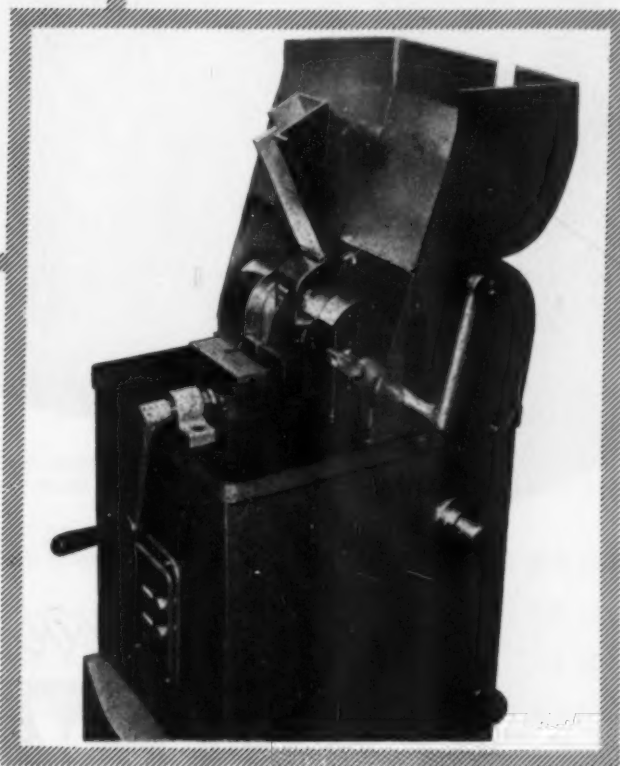
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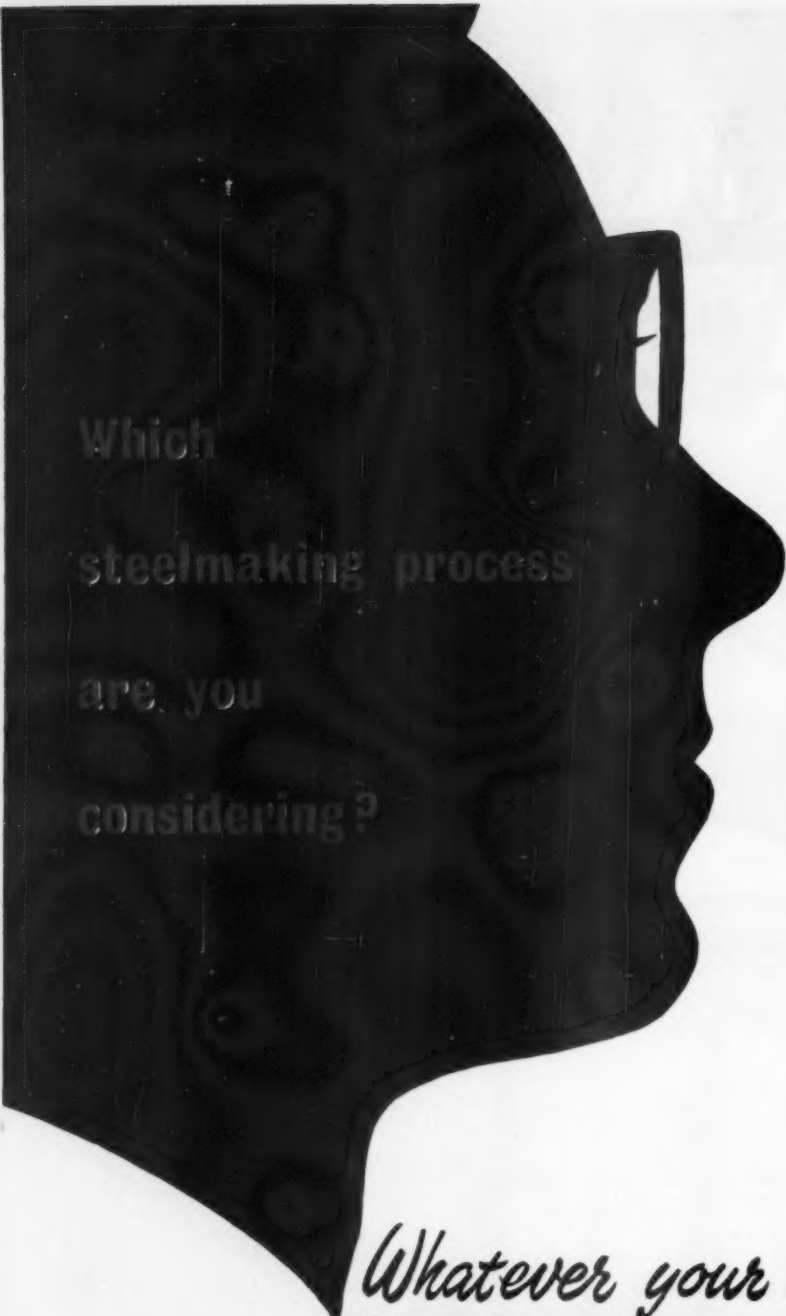
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THE BRITISH JOURNAL OF METALS  
INCORPORATING THE METALLURGICAL ENGINEER

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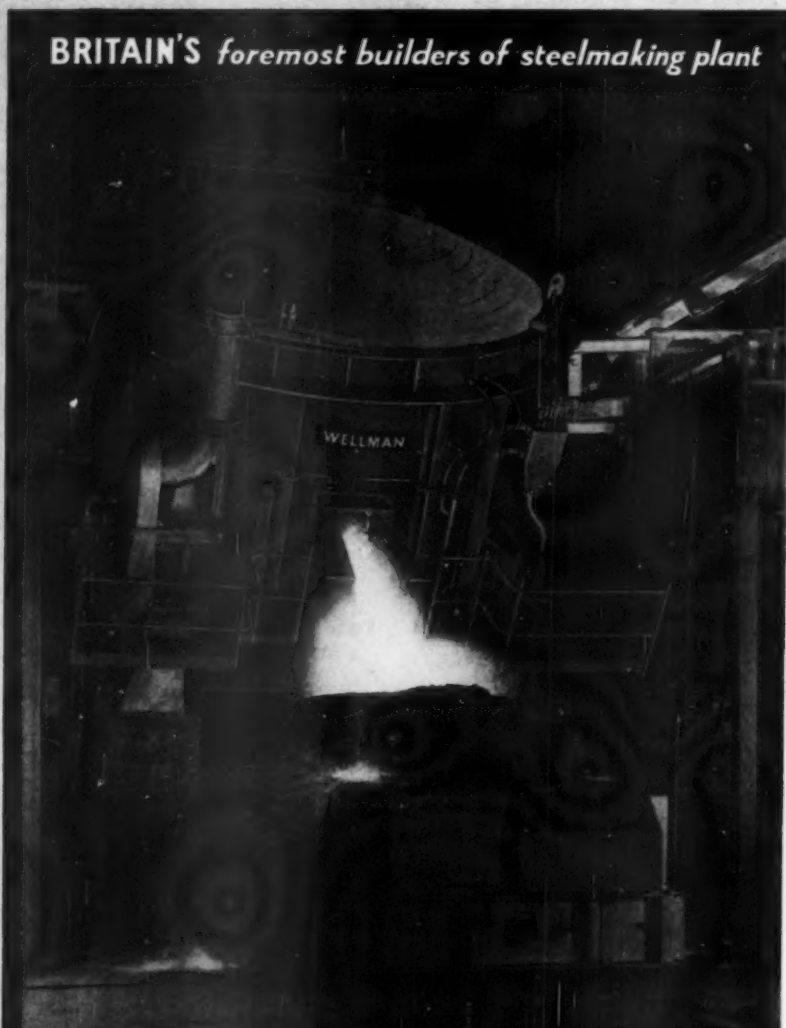
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THE BRITISH JOURNAL OF METALS  
INCORPORATING THE "METALLURGICAL ENGINEER"

SEPTEMBER, 1960

Vol. LXII. No. 371

## The First Twenty-five Years

IN a recent issue of *Steel Review*, the British Iron and Steel Federation's quarterly publication, Sir Ellis Hunter, chairman and managing director of Dorman Long and Co., Ltd., and president of the Federation during the important post-war period from 1945 to 1953, reviews the first twenty-five years of the Federation's existence—from 1934 to 1959. The setting-up of the Federation was an outcome of the difficult decade preceding 1934, when the steel industry "had never had it so bad." At this time the continental steel producers were protected by considerable import tariffs which enabled them to regulate prices in their home and export markets and to dump surplus output at prices below the cost of production in the large unprotected market in this country. Without protection the British industry could neither compete nor retaliate, and in the home market consumers such as those producing capital goods were facing such intense competition that they were forced to buy much of their steel at the lowest level—the dumping price of Continental producers. In these circumstances most of the co-operative associations in the industry ceased to function, price-cutting drastically reduced profits, many companies were subjected to financial reconstruction involving the writing-off of much capital, unemployment was rife, and the development of the industry came to a halt.

From 1925 onwards, Sir William Larke, Director of the National Federation of Iron and Steel Manufacturers, led a ceaseless agitation for an import tariff, without which stability in the industry could not be achieved, nor could the much needed re-equipment be undertaken. Following the worldwide economic depression of 1929 to 1931, and the return of the National government in October 1931, a 10% import duty was imposed on all manufactured goods and the Import Duties Advisory Committee was set up to consider where additional protection was at least temporarily necessary.

The Committee recommended that special protection should be temporarily afforded the steel industry on condition that it took immediate steps to reorganise itself under the aegis of a strong central organisation, the idea being that the industry should embark on a programme of re-equipment and expansion, and should put itself into a position to negotiate with the European steel cartel in order to limit imports into this country and to secure a fair share of world exports.

The call for a strong central organisation was met by the formation of the British Iron and Steel Federation, which was designed to have much greater influence than the existing National Federation of Iron and Steel Manufacturers. The constitution of the new Federation effected a considerable transfer of responsibility from the associations concerned with particular products and

regions to the executive committee of the Federation. Perhaps the most important feature of the constitution was the provision for the appointment of a man of high standing, but without an interest in any steel company, as independent chairman of the executive committee. The industry was extremely fortunate in that its search for a suitable man resulted in the appointment of Sir Andrew Duncan, a man of outstanding personality with an exceptionally varied experience of large-scale industry.

Under Sir Andrew's guidance the steel companies, freed from the struggle for survival, were able to approach national problems in a more co-operative spirit, and in 1936 agreed to declare their intentions on future developments to the Federation. Agreements on both imports and exports were negotiated with the cartel, and to avoid any possible accusation of advantage being taken of the protection of the home market contrary to the national interest, the industry instituted a scheme of voluntary submission to the Import Duties Advisory Committee of any proposals for adjustment of prices and for major developments. A further example of co-operative action was the establishment of the British Iron and Steel Corporation to import scrap, pig iron and semi-finished steel on behalf of the industry.

By 1939 the situation in the industry had been transformed, outstanding development schemes completed including the new steel and tube works at Corby, the new heavy steel works at Cardiff, and the continuous wide strip mills at Ebbw Vale and Shotton. The outbreak of war interrupted development but with Government encouragement the Federation took active steps in 1944 to shape the industry's post-war programme. The result was that a comprehensive development plan was laid before the new Labour Government in December 1945, and approved by it. This was the first development plan ever conceived by an industry as a whole, and it was remarkable both in its scope and in the speed with which it was completed. Since that first programme, others have been drawn up and implemented, in the course of which the capacity of the industry has been doubled.

Further examples of co-operative action in the post-war steel industry are to be found in the fields of research, raw materials, and training. Research on behalf of the industry as a whole had been undertaken for some years, but in order to co-ordinate and expand these co-operative activities it was decided to establish and finance the British Iron and Steel Research Association which is now the largest industrial research association in the country. In the raw materials field it was agreed that ore should be imported centrally on behalf of the industry by BISC (Ore), Ltd.—a new subsidiary of the British Iron and Steel Corporation—in order to ensure adequate imports in the difficult supply and shipping conditions anticipated. As the last example, a central training committee and area training committees were



established to encourage the further development of training facilities in the industry, and, in particular, to make available to the smaller companies the knowledge and experience of the large companies.

No account of the activities of the Federation would be complete without reference to nationalisation, the threat of which must have absorbed a considerable proportion of the Federation's efforts from time to time during the last fifteen years. Throughout this period the Federation has effectively presented the industry's case to the Government, on the one hand, and the general public on the other. Although the industry was nationalised for a period of eight months, in practice the Federation continued to co-ordinate the activities of the companies of which the majority—all the smaller ones—were never nationalised. In September, 1946, the Government set up an Iron and Steel Board, which continued to operate until March 1949, and provided a remarkable demonstration of successful public control, in full co-operation with all branches of the industry. During 1947 the Federation and Cabinet representatives reached an understanding providing for giving the Board additional powers and functions to counter any sugges-

tions that it could only act negatively. These discussions were terminated when the Government decided to proceed with nationalisation of the industry, and with the announcement of that decision the Board ceased to function. The Iron and Steel Act, 1953, created a new supervisory Iron and Steel Board, provided this time with statutory powers. In essence, this was what had been agreed with the representatives of the Labour Government in 1947.

In concluding his survey, Sir Ellis points out that the wheel has now turned full circle, in that with the passage of the Restrictive Practices Act and the formation of the European Free Trade Association the primary accent is again on competition. There is, however an important difference. In the 1920's Britain alone adhered to free trade at a time when her competitors barricaded themselves behind high tariffs. Today, the industry has some safeguards against dumping, and competition is on more equal terms. Even more important, the complete re-equipment of the industry and the doubling of its capacity made possible by the changes inaugurated in 1932-34 has revolutionised the industry's competitive position.

## Personal News

DR. J. C. HUDSON, who has been in charge of the British Iron and Steel Research Association's research on corrosion for the last fifteen years—ever since the formation of the Association in 1945—retired at the end of August. He continues to have very close ties with the Association, however, since he has agreed to act as consultant to both the Corrosion Advice Bureau and the Chemistry Department. Following a period with the Atmospheric Corrosion Sub-Committee of the British Non-Ferrous Metals Research Association, Dr. Hudson became, in 1929, investigator to the newly formed Corrosion Committee of The Iron and Steel Institute, since taken over by B.I.S.R.A., and has continued in this post until now. He has also served as Head of B.I.S.R.A.'s Corrosion Advice Bureau, since its formation in 1954. Dr. Hudson has recently received two major awards in recognition of his outstanding contributions to corrosion engineering and ferrous metallurgy—the Frank Newman Speller Award for 1959 from the American National Association of Corrosion Engineers, and the Sir Robert Hadfield Bronze Medal for 1960 from the Iron and Steel Institute.

MR. W. MCK. WRIGHT has been appointed resident area sales manager in the Middle East for Armstrong Whitworth (Metal Industries), Ltd. Mr. Wright has lived in the Middle East, India and Pakistan for twenty-seven years and for the past three has been representing a group of companies in the Middle East. His address is: Imm. Almawi, Rue Bliss, Ras Beirut, Beirut, Lebanon. Another appointment announced by the company is that of Mr. G. CHESHAM as special representative to the Soviet bloc of countries. Mr. Chesham, whose address is: Wien 1, Ander Hulben 1/12A, Austria, is a former Machine Tool Trade Association official with an intimate knowledge of East/West trade procedure.

THE United Steel Cos., Ltd., announce with regret the resignation of Mr. A. JOLLIE from his position as director and general manager of Steel, Peech and Tozer on July 31st. MR. T. S. KILPATRICK, at present director

and general manager of Workington Iron and Steel Co., has been appointed director and general manager of Steel, Peech and Tozer from August 1st.

MR. T. H. KELSEY, whose appointment as assistant general manager of the Witton Engineering Works of The General Electric Co., Ltd., was announced in October 1958, has now been appointed deputy general manager to Mr. J. J. GRACIE, director of the company, and general manager of Witton Engineering Works.

MR. C. M. STOCH, head of the engineering section of the British Steel Castings Research Association, Sheffield, recently returned to this country from a tour of steel foundries and metallurgical research institutes in Czechoslovakia and Poland.

MR. F. BAILLIE, who joined the Cambridge Instrument Company as production manager on August 2nd, 1960, will be responsible for all aspects of production planning at the company's three factories in the United Kingdom. Mr. Baillie was formerly general manager at the Greenock factory of IBM (United Kingdom), Ltd. Prior to joining IBM in 1955, he spent a year with Muirhead and Co., Ltd., and, before that, several years with Kelvin and Hughes, Ltd.

MR. R. WALLER has been appointed mechanical maintenance superintendent at Steel, Peech and Tozer, a branch of The United Steel Cos., Ltd. He succeeds Mr. A. STARK who has retired after 22 years' service. Mr. Waller joined the drawing office staff of Steel, Peech and Tozer in 1933, and became new construction engineer in 1953. He has been assistant mechanical maintenance superintendent since 1959.

THE following appointments have been made in the sales organisation of The Brightside Foundry and Engineering Co., Ltd., of Ecclesfield, Sheffield: MR. G. E. ROBINSON, general sales manager; MR. G. H. LEWIS, sales manager, foundry division; and MR. T. HESSEY, technical sales manager.

MR. D. R. WARD JONES who recently became general manager of United Steel's Ore Mining Branch has been appointed a director of United Coke and Chemicals Co., Ltd.



# The Effect of Cold Work on the Fatigue Properties of Two Steels

By N. E. Frost, B.Sc.(Eng.), A.M.I.Mech.E.

National Engineering Laboratory, East Kilbride

*Fatigue tests have been carried out on blanks of typical mild and nickel-chromium steels after subjecting them to varying amounts of cold work, in order to determine the maximum possible increase in fatigue strength resulting solely from cold work. An appreciable increase in fatigue limit can be achieved by cold working mild steel, there being an optimum value for the amount of prior strain. There is some indication that the fatigue limit of a 2½% nickel-chromium alloy steel may be slightly increased by cold work*

THE effect of cold work on the plain fatigue properties of a material varies with the material. For example, mild steel and titanium both show an increase in fatigue strength with moderate amounts of cold work. Copper and copper alloys, although showing an increase in tensile strength with cold work, exhibit very little, if any, corresponding increase in fatigue strength, while high strength wrought aluminium alloys may have their fatigue properties reduced by quite moderate amounts of cold work.<sup>1-4</sup>

If a notched fatigue specimen is statically pre-loaded so that plastic deformation occurs at the notch root, residual stresses of opposite sign to the applied external loading will remain on unloading. In addition, the plastically deformed material may work harden. The subsequent fatigue strength of such pre-loaded notched specimens will depend on both the fatigue strength of the work hardened material at the notch root and the value of the induced residual stresses, compressive residual stresses being beneficial and tensile residual stresses detrimental. Materials, such as mild steel, which exhibit relatively large non-elastic cyclic strains under the imposed cyclic loading are able to relax the residual stresses so that they may not greatly affect the subsequent fatigue strength. Dugdale,<sup>5</sup> for example, showed that statically pre-loading mild steel notched specimens had little effect on the subsequent notched fatigue strength, but for nickel steel notched specimens, pre-compressing resulted in considerably lower and pre-stretching considerably higher notched fatigue strengths.

Thus, it might be expected that the notched fatigue strength will be dependent on the machining processes used to form the notch, as different processes will result in varying degrees of work hardening of the material at the notch root and induce different values of residual stresses. For mild steel notched specimens the induced

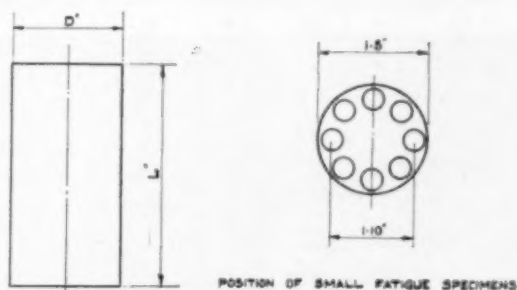


Fig. 1.—Blank for compression tests.

residual stresses may have little effect, but Frost<sup>1</sup> has shown that for moderate amounts of cold work the fatigue strength of the material increases, approximately linearly, with the amount of cold work. On the other hand, with, say, high strength alloy steel notched specimens, the induced residual stresses may be important whereas little increase in the fatigue strength of the material may result from cold work.

To determine the maximum possible increase in fatigue strength resulting solely from cold work, blanks of typical mild and nickel-chromium steels have been subjected to varying amounts of cold work and the fatigue strength of specimens cut from the worked blanks obtained.

Some results, although irrelevant to the main purpose of the work, are included in the paper as they may be of general interest.

## Experimental Details

The chemical analysis and mechanical properties of the bar materials used are set out in Table I. All blanks and specimens were cut with their longitudinal axes parallel to the longitudinal axis of the bar material. Blanks of the two materials were cold worked by applying either a compressive or torsional plastic pre-strain. In the former case, cylindrical blanks of the form shown in Fig. 1 and having the dimensions given in Table II were used, these being compressed in a 200-ton capacity hydraulic test machine, while for applying torsional pre-strain the blanks were of the form shown in Fig. 2, these being twisted in a 60,000-in. lb. capacity torsion machine. The various amounts of compressive strain and torsional twist applied are given in Tables II and III. All nominal

TABLE I.—DETAILS OF BAR MATERIALS USED FOR TESTS

Material	Chemical Analysis	Condition	Tensile Strength (tons/sq. in.)	Yield stress (tons/sq. in.)	Elongation (% on 2 in.)
Mild Steel	0.15% C 0.45% Mn 0.014% S 0.015% P	Normalized and straightened	29	23	30
Nickel-Chromium Steel	0.45% C 2.64% Ni 0.75% Cr 0.65% Mn 0.58% Mo 0.012% S 0.012% P	oil quenched 850° C., air cooled 650° C.	63	54	24

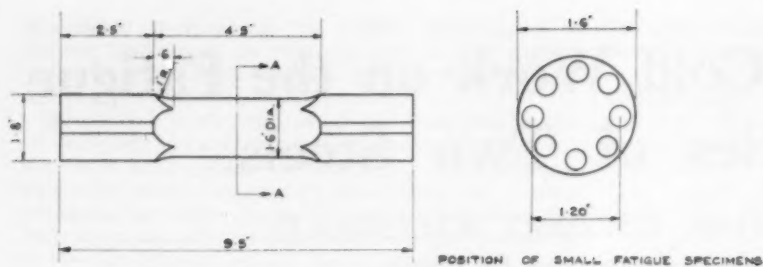


Fig. 2.—Static torsion specimen.

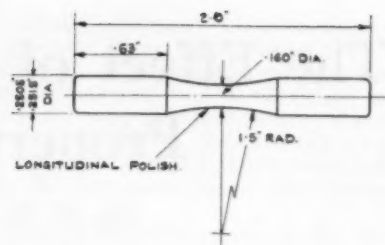


Fig. 3.—Small rotating bending fatigue specimen.

TABLE II.—DETAILS OF COMPRESSION BLANKS

Blank No.	Blank Dimensions (in.)			Nominal Plastic Strain (%)	Nominal Stress (tons/sq. in.)
	Original Diameter	Original Length	Final Length		
Mild Steel					
TON 8F2	1.50	3.5	2.75	21	53
TON 8K1	1.50	3.5	1.80	49	100
TON 8K2	1.50	3.5	1.80	49	100
TON 8F1	1.50	3.5	1.80	49	100
TON 1R <sup>a</sup>	1.50	4.1	1.40	66	—
TON 7 <sup>a</sup>	3.00	7.0	—	—	—
	1.60	—	1.20	83	—
	3.00	10.0	—	—	—
TON 5 <sup>a</sup>	1.50	—	1.40	86	—
Nickel-Chromium Steel					
RSU 9N	1.50	4.5	3.80	16	89
RSU 9M	1.50	3.0	2.30	23	100
RSU 5 <sup>a</sup>	1.50	2.0	—	—	—
	1.25	—	1.80	55	—

<sup>a</sup> Compressed up to the maximum capacity of the test machine, i.e., 200 tons. Diameter then decreased and specimen relieved.

$$\text{Nominal plastic strain \%} = \frac{\text{original length} - \text{final length}}{\text{original length}} \times 100$$

$$\text{Nominal stress} = \frac{\text{final load}}{\text{original area}}$$

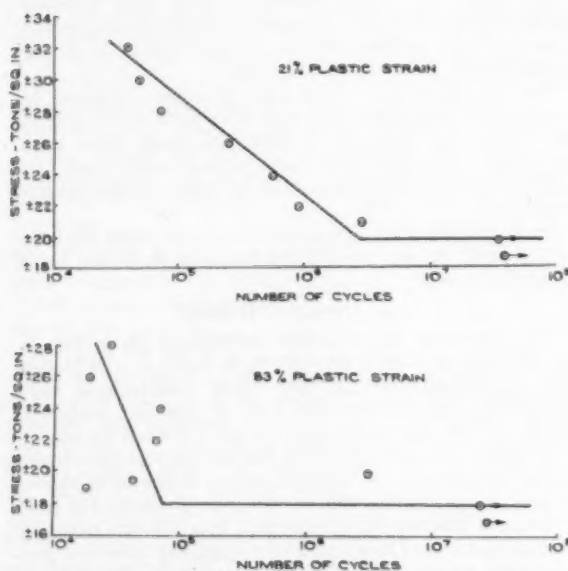


Fig. 4.—Typical S/N curves for specimens cut from mild steel blanks subjected to various nominal compressive plastic strains.

TABLE IV.—FATIGUE LIMITS OF MILD STEEL SPECIMENS

Test Mark	Prior Cold Work	Vickers Hardness Number	Fatigue Limit (tons/sq. in.)
Pre-loaded in Compression			
	Nominal Compressive Plastic Strain (%)		
TON 1Z and J	0	146	± 16½
TON 1M	7	180	± 17
TON 8F2	21	207	± 20
TON 3M	33	222	± 20½
TON 8K1	49	232	± 22½
(Stress relieved for 1 hour at 800° C. in vacuo)			
(TON 8F1)	49	116	± 13
(Stress relieved for 1 hour at 650° C. in vacuo)			
(TON 8K2)	49	126	± 13½
(Stress relieved for 1 hour at 400° C. in vacuo)			
(TON 8F1)	49	201	± 20
(Stress relieved for 1 hour at 200° C. in vacuo)			
(TON 8F1)	49	217	± 21
TON 1R	66	224	± 17
TON 7	83	232	± 18
(Stress relieved for 1 hour at 650° C. in vacuo)			
(TON 7)	83	108	± 11
TON 5	86	232	± 17
(Stress relieved for 1 hour at 650° C. in vacuo)			
(TON 5)	86	107	± 11
Pre-loaded in Torsion			
	Plastic Twist Over Gauge Length (deg.)		
TON 1Z and J	0	146	± 16½
TON 8L	90	201	± 18
TON 8G	180	220	± 20½
TON 8E	360	240	± 21½
TON 8C	630	259	± 22

strains and angles of twist were obtained from measurements of the blanks after removal from the testing machine and, consequently, represent permanent plastic

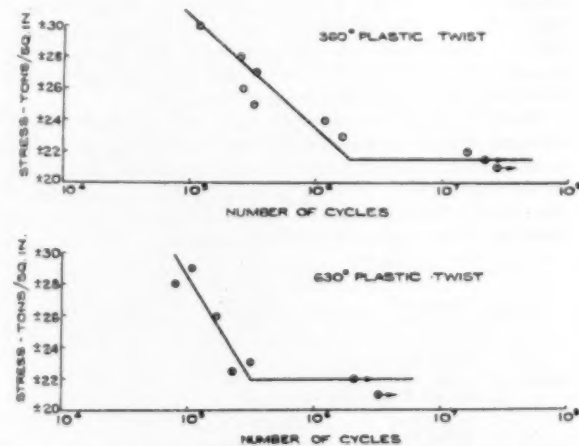
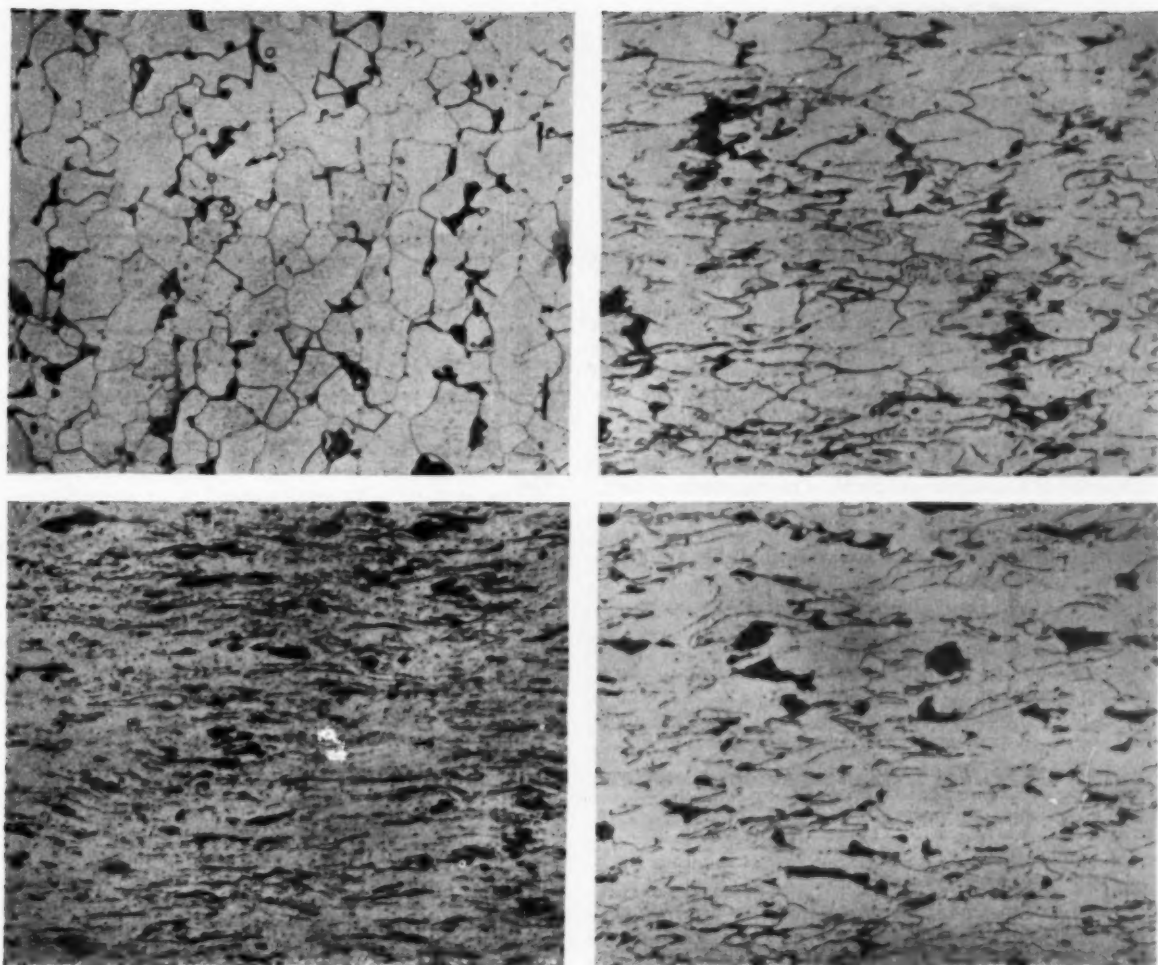


Fig. 5.—S/N curves for specimens cut from torsional pre-strained mild steel blanks.



(a) TON 1J—as machined  
(b) TON 8K1—49% nominal compressive pre-strain  
(c) TON 5—86% nominal compressive pre-strain  
(d) TON 8C6—630° plastic twist

Fig. 6.—Photomicrographs of pre-strained mild steel specimens.

× 200

deformation. With some of the compression blanks the required amount of plastic deformation could not be obtained in a single loading. In such cases the blank was loaded to the machine capacity, then removed from the machine and the outside diameter machined to a smaller size. The blank was then again compressed, either to the required deformation or to the machine capacity, in which case the procedure was repeated.

Nine fatigue specimens, 0.16-in. diameter, of the form shown in Fig. 3, were cut longitudinally out of each deformed blank from the position indicated in Figs. 1 and 2. These were tested in a cantilever rotating-bending machine running at about 4,000 rev./min. Vickers diamond pyramid hardness tests were carried out on the ends of the fatigue specimens.

### Experimental Results

#### Mild Steel

The fatigue limits obtained on specimens cut from blanks which had been subjected to nominal compressive plastic strains of 21%, 49%, 66%, 83% and 86% are given in Table IV and typical S/N curves are shown in Fig. 4. The results obtained previously by the author<sup>1</sup> on blanks of identical material compressed to nominal plastic strains of 0, 7% and 33% are also given in Table IV. Similarly, the fatigue limit of specimens cut from blanks pre-strained in torsion to give angles of plastic

TABLE III.—DETAILS OF TORSION BLANKS

Blank No.	Blank Diameter (in.)	Plastic Twist Over Gauge Length (deg.)
Mild Steel		
TON 8E	1.6	360
TON 8C	1.6	630
Nickel-Chromium Steel		
RSU 16H	1.6	360

Angle of twist at fracture is 630° and 400° for the mild steel and nickel-chromium steel respectively.

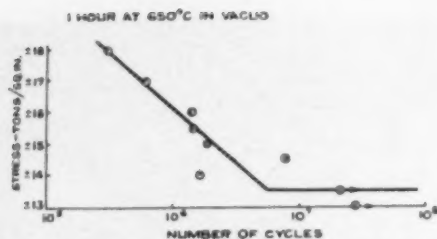


Fig. 7.—S/N curve for specimens cut from pre-compressed mild steel blank then stress-relieved.

twist over the gauge length of 360° and 630° are given in Table IV together with those obtained previously for twists of 90° and 180°. Typical S/N diagrams are given in Fig. 5. A selection of photomicrographs of the pre-strained mild steel specimens are shown in Fig. 6.

To investigate the effects of stress relieving, four batches of specimens were cut from blanks that had been subjected to a nominal compressive plastic strain of 49%. They were heat treated for 1 hour *in vacuo* at 200° C., 400° C., 650° C. and 800° C., respectively, prior to testing. The fatigue limits obtained are given in Table IV, and a typical S/N diagram is shown in Fig. 7.

#### Nickel-Chromium Steel

The fatigue limits obtained on specimens cut from blanks which had been subjected to nominal compressive plastic strains of 0, 16%, 23% and 55%, or given a plastic twist, over the gauge length, of 360°, are given in Table V. Two typical S/N curves are shown in Fig. 8.

TABLE V.—FATIGUE LIMITS OF NICKEL-CHROMIUM STEEL SPECIMENS.

Test Mark	Prior Cold Work	Vickers Hardness Number	Fatigue Limit (tons/sq. in.)
Pre-loaded in Compression			
	Nominal Compressive Plastic Strain (%)		
RSU 12C	0	320-335	± 35
RSU 9N	16	335-345	± 35
RSU 9M	23	335-355*	± 39
RSU 5	55	330-365	± 38
		360-380*	
Pre-loaded in Torsion			
	Plastic Twist over Gauge Length (deg.)		
RSU 12C	0	320-335	± 35
RSU 16H	360	360-375	± 36

\* Heat treated 1 hour at 600° C. *in vacuo*.

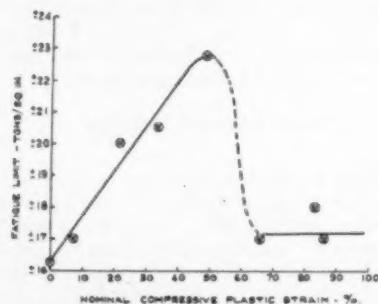


Fig. 9.—Fatigue limit v. nominal compressive plastic strain (mild steel).

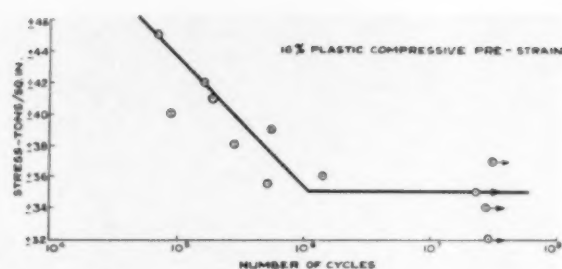


Fig. 8.—S/N curves for specimens cut from pre-compressed nickel-chromium steel blanks.

A few fatigue specimens were stress relieved for 1 hour at 600° C. *in vacuo* before testing.

The average Vickers hardness number for each series of specimens is also included in Tables IV and V.

#### Discussion

Considering first the mild steel results, the fatigue limits are plotted against the corresponding nominal compressive plastic strain or plastic twist in Figs. 9 and 10, respectively. In the former case, the fatigue limit increases linearly with increase in nominal plastic strain from  $\pm 16\frac{1}{2}$  tons/sq. in. to  $\pm 22\frac{1}{2}$  tons/sq. in. at about 50% nominal strain. Higher values of nominal strain cause a sharp decrease in fatigue limit to about  $\pm 17$  tons/sq. in. In addition to the sudden decrease in fatigue limit, the finite life portion of the S/N curves becomes very much steeper, especially so for the 83% and 86% nominal pre-strain tests, fractures tending to occur before  $10^5$  cycles or not at all.

For the specimens pre-strained in torsion, the fatigue limits appear to increase continually with initial plastic twist from a value of  $\pm 16\frac{1}{2}$  tons/sq. in. up to a maximum

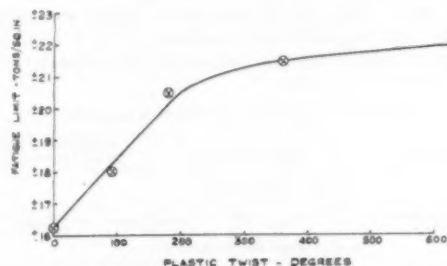


Fig. 10.—Fatigue limit v. plastic twist (mild steel).



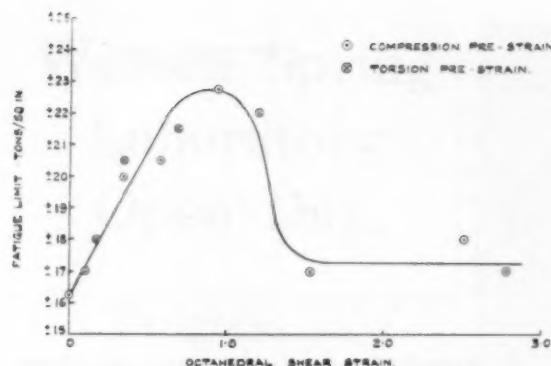


Fig. 11.—Fatigue limit v. prior octahedral shear strain.

value of  $\pm 22$  tons/sq. in. at an angle of twist of  $630^\circ$ . The relationship is linear up to about  $180^\circ$  plastic twist corresponding to a fatigue limit of  $\pm 20$  tons/sq. in. Although no decrease in fatigue limit occurred the finite portion of the S/N curve for the  $630^\circ$  plastic twist specimens was very steep, as found for the two high pre-compressive strain series of tests.

If the compressive and torsion pre-strains are transformed to equivalent octahedral shear strains, the results may be plotted on a single diagram. The octahedral shear strain is given by:

$$\epsilon_{oct} = \frac{2}{3} \sqrt{(\epsilon_1 - \epsilon_2)^2 + (\epsilon_2 - \epsilon_3)^2 + (\epsilon_3 - \epsilon_1)^2}$$

where  $\epsilon_1$ ,  $\epsilon_2$  and  $\epsilon_3$  are principal strains. For compression this gives:

$$\epsilon_{oct} = \sqrt{2} \epsilon_1$$

and for torsion:

$$\epsilon_{oct} = \sqrt{3} a$$

where  $a = \frac{r\theta}{l}$

$r$  being the effective specimen radius,  $\theta$  the angle of twist, and  $l$  the specimen gauge length. For large compressive strains it is more satisfactory to use natural rather than nominal strains, i.e.,

$$\text{if nominal strain } \epsilon_1 = \frac{l_0 - l}{l_0}$$

$l_0$  being the original gauge length and  $l$  the final gauge length, then

$$\text{natural strain} = \ln \frac{1}{1 - \epsilon_1}$$

For torsional strains, the differences between nominal

TABLE VI.—FATIGUE LIMITS AND PRIOR OCTAHEDRAL SHEAR STRAINS FOR MILD STEEL SPECIMENS

Test Mark	Fatigue Limit (tons/sq. in.)	Vickers Hardness Number	Octahedral Shear Strain
TON 1Z and J	$\pm 16\frac{1}{2}$	146	—
TON 1M	$\pm 17$	180	0.103
TON 8L	$\pm 18$	201	0.171
TON 8G	$\pm 20\frac{1}{2}$	290	0.342
TON 8F2	$\pm 20$	207	0.340
TON 3M	$\pm 20\frac{1}{2}$	225	0.577
TON 8E	$\pm 21\frac{1}{2}$	240	0.684
TON 8K1	$\pm 22\frac{1}{2}$	232	0.938
TON 8C	$\pm 32$	259	1.196
TON 1B	$\pm 17$	224	1.526
TON 7	$\pm 18$	232	2.505
TON 5	$\pm 17$	232	2.780

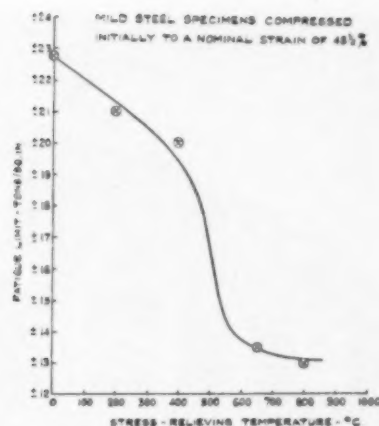


Fig. 12.—Effect of stress-relieving temperature on fatigue limit.

and natural strain are much smaller; for the maximum twist of  $630^\circ$ , the difference is about 7%.

The values of octahedral shear strain and the corresponding values of fatigue limit and hardness number are given in Table VI, the fatigue limits being plotted against prior octahedral strain in Fig. 11. It is seen that on a basis of equivalent octahedral shear strain fair correlation is obtained between the two methods of cold working, the maximum fatigue limit occurring at an octahedral shear strain of about 1.0. Higher strains cause a drastic reduction in fatigue strength, to little over that for zero pre-strain, and this might well be due to the formation of sub-microscopic cracks, as all tests on blanks strained beyond the optimum value resulted in S/N diagrams having steep finite life portions.

The hardness values also increase with prior cold work, attaining a maximum value of about 230–240 Vickers hardness at about the same strain value as that at which the maximum fatigue limit occurs. However, further cold work does not alter the hardness value appreciably, there being no drastic decrease in value comparable to the decrease in fatigue limit. Hardness values thus give no definite indication when the optimum amount of prior cold work for maximum fatigue strength is exceeded.

The fatigue limits of compressed and stress-relieved specimens are plotted in Fig. 12. The fatigue limit is seen to decrease as the stress relieving temperature is increased, a rapid drop occurring between  $400^\circ\text{C}$ . and  $650^\circ\text{C}$ ., only a slight further reduction occurring at  $800^\circ\text{C}$ . The fatigue limit of the fully stress-relieved specimens is some  $\pm 3$  tons/sq. in. below that for the original bar material. This is due both to initial cold work in the bar material and to work hardening of the surface layers of the fatigue specimens due to machining and polishing. Thus the rotating-bending fatigue limit of mild steel can be increased from  $\pm 13\frac{1}{2}$  tons/sq. in. for the fully stress-relieved condition to  $\pm 22\frac{1}{2}$  tons/sq. in., at some optimum value of cold work.

A few of the specimens which had been subjected to the severest prior cold work (i.e., 83% and 86% nominal compressive strain) were stress relieved at  $650^\circ\text{C}$ . for 1 hour *in vacuo* prior to testing. The finite life portion of the resulting S/N diagram now had a slope comparable to that for virgin specimens, but the fatigue limit decreased to  $\pm 11$  tons/sq. in., i.e., about  $\pm 2$  tons/sq. in.

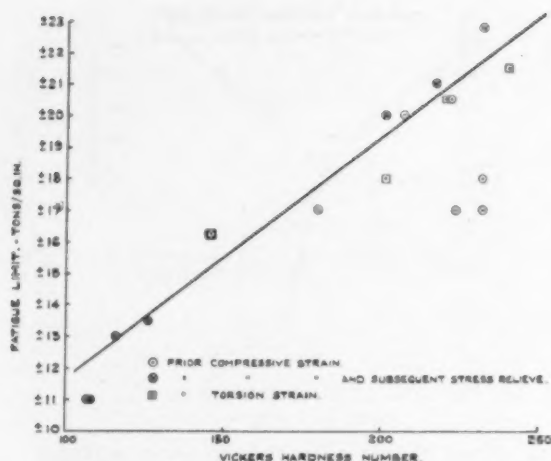


Fig. 13.—Hardness v. fatigue limit (mild steel).

lower than the similarly stress-relieved specimens previously discussed. The hardness values also decreased.

The Vickers hardness values are plotted against the fatigue limit for all the mild steel specimens in Fig. 13. As long as the prior cold work is below the optimum value for maximum fatigue strength, then, irrespective of method of cold working or whether the material is subsequently stress relieved, the fatigue limit is proportional to the hardness. However, for excessive amounts of cold work the hardness is not indicative of the fatigue strength. In such cases it would appear that incipient micro-cracks are formed, the fatigue limit of  $\pm 17$  tons/sq. in. being equal to the critical propagation stress necessary for their subsequent growth. The effect of stress relieving is to reduce the propagation stress to a value of  $\pm 11$  tons/sq. in. It is interesting to note the very low hardness value, i.e., 107 Vickers hardness obtained on stress relieving these latter specimens as compared to a value of 126 Vickers hardness for similarly stress-relieved material not critically cold worked.

Turning now to the alloy-steel results, there appears to be little change in fatigue limit due to cold working by either prior compressive or torsional pre-strain, the values varying between  $\pm 35$  tons/sq. in. and  $\pm 39$  tons/sq. in. However, as with the mild steel, those specimens which had been the most severely cold worked gave an S/N curve with a very steep finite life portion. The fatigue limits are plotted against octahedral shear strain in Fig. 14, and although there is some tendency for the fatigue limit to increase with the amount of prior cold work, the relationship is rather erratic. This erratic behaviour is reflected in the hardness values which showed considerable scatter, although again generally tending to increase with amount of cold work. A few fatigue specimens were stress relieved for 1 hour at 600° C. *in vacuo* before testing. These tended to show a slight improvement in fatigue strength over comparable specimens, which had not been heat treated, this being compatible with the corresponding increase in hardness that occurred.

#### Conclusions

(1) The effect of prior cold work, by either compressive or torsional plastic deformation, can increase the fatigue limit of a fully stress-relieved mild steel from  $\pm 13\frac{1}{2}$  tons/sq. in. to a maximum value of  $\pm 22\frac{1}{2}$  tons/sq. in.

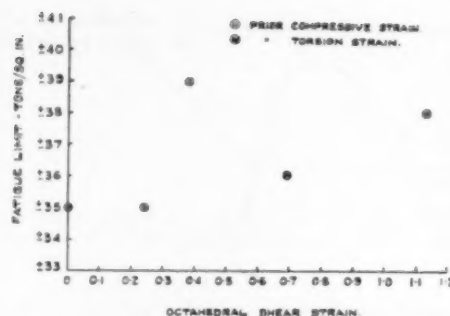


Fig. 14.—Fatigue limit v. prior octahedral shear strain (nickel-chromium steel).

(2) There is an optimum value of the amount of prior plastic strain at which the maximum fatigue limit occurs. Further prior strain results in a considerable drop in the fatigue limit but does not greatly affect the hardness value.

(3) There is some indication that the fatigue limit of a 2½% nickel-chromium alloy steel (En 26) may be slightly increased by cold work.

#### Acknowledgments

The work has been carried out as part of the research programme of the National Engineering Laboratory of the Department of Scientific and Industrial Research and the paper is published by permission of the Director of the Laboratory.

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#### Stainless Steel Tuyère Blowpipes

A SUPPLY of centrispun tuyère blowpipes has recently been produced and delivered by Firth-Vickers Stainless Steels, Ltd., to Appleby-Frodingham Steel Company of Scunthorpe. These have been fitted to the "Queen Bess", one of four blast furnaces at the company's South Ironworks, where they will be used for injecting preheated air at the rate of 85,000–90,000 cu. ft./min. at a temperature of 750° C. and a pressure of 30 lb./sq. in. It is expected that the output of the "Queen Bess" will be of the order of 8,500–9,000 tons per week, but during the course of the next two years it is anticipated that the working temperature will be stepped up to 900° C. with a subsequent production increase.

Centrispun blowpipes produced in Firth-Vickers H.R. Crown 1 steel have been used successfully in the industry for more than three years and are now in regular production in the Firth-Vickers centrispinning foundries. The original blowpipes produced in this material were fabricated from four individual spinnings but Firth-Vickers, who pioneered the centrispinning of stainless steels, have since developed new techniques which have made it possible to produce a blowpipe as a one-piece spinning from a special die. Past experience has proved that blowpipes manufactured from this material give many times the service life of those produced in mild steel or haematite iron and ultimately reduce replacement and maintenance costs.

# Warren Spring Laboratory Open Day

## Progress in Mineral Processing Research



The pilot-scale mineral processing laboratory houses plant suitable for the treatment of ores at rates up to 1,000 lb. per hour.

THE Warren Spring Laboratory at Stevenage, newest of the D.S.I.R.'s research establishments, has, since it was officially opened by Lord Hailsham, Minister of Science, in June last year, embarked on a versatile programme of research. This is in keeping with the Research Council's original plans for the Laboratory as one that should be ready to carry out process research and development over a wide field, and to include work on any subjects of national importance (especially involving pilot scale investigations) which cannot be fitted into the programme of another research body. The Warren Spring Laboratory replaced the Fuel Research Station on 1st January, 1959, and it was agreed by the Research Council that only two sections of the research programme of that Station should be continued at the new Laboratory, namely atmospheric pollution and the synthesis of oils. Such a decision does not, however, preclude the Laboratory from the investigation of other fuel research problems; in fact, Council considers that it should tackle fuel problems in the national interest which are not being studied elsewhere, and are not appropriate to other existing organisations engaged in fuel research.

### Round the Divisions

The main research divisions (Atmospheric Pollution, Chemical Engineering, Process Development, and Mineral Processing) are supported by two service divisions (Physical and Chemical Services and Intelligence and Engineering Services). Before considering in rather more detail the Mineral Processing Division, brief reference may be made to the work in progress in the other divisions.

#### *Chemical Engineering and Process Development*

Taking Chemical Engineering and Process Development together, the objective is the provision of information that will permit industry to design full-scale processing plant with greater accuracy and reliability and to develop new equipment and processes. One item of the present programme is the study of gas-liquid systems designed to obtain data that will permit

more accurate prediction of the performance of gas-liquid contacting equipment, and thus facilitate the design of distillation columns, gas absorption towers, and reaction vessels of the liquid-phase type, of which the Fischer-Tropsch "slurry" reactor is a particular example.

A study is also being made of dropwise condensation promoters, aimed at determining the mechanism of attachment of synthetic promoters to metal surfaces in order to facilitate the development of compounds which would be effective for prolonged periods of use with a wide range of metal surfaces.

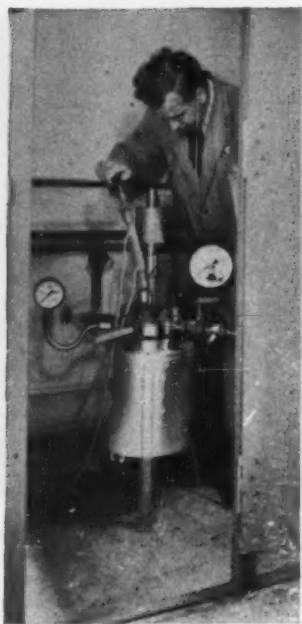
A process under investigation is the Fischer-Tropsch synthesis, in which a mixture of carbon monoxide and hydrogen (which can be produced from low grade coal) is converted into gaseous and liquid fuels and chemical products. The aim of the work, which has been undertaken at the request of the Ministry of Power, is to perfect the development of a liquid-phase system for carrying out this process. The two main problems requiring solution are the development of a suitable catalyst, and the design and operation of a suitable type of reaction vessel. Basic research on catalysts and reaction mechanism is also being carried out with a view to effecting further improvements in the process.

It was the intention in setting up the Warren Spring Laboratory that it should be available to undertake sponsored research work, and in these divisions development work is being sponsored on fuel cells, agitation equipment, demisters, and specific problems in chemical engineering.

#### *Atmospheric Pollution*

The work of the Atmospheric Pollution Division is designed to assist in achieving clean air. Part of the work is therefore concerned with methods of reducing air pollution, such as preventing the emission of smoke from diesel lorries, removing oxides of sulphur from chimney gases, and improving the dispersion of chimney gases. The other part consists of mapping the distribution of pollution throughout the country so that existing clean air legislation may be applied in the best





**An autoclave in the Mineral Processing Division**

possible way and the value of each ameliorative action assessed, taking full account of the effects of weather and local conditions.

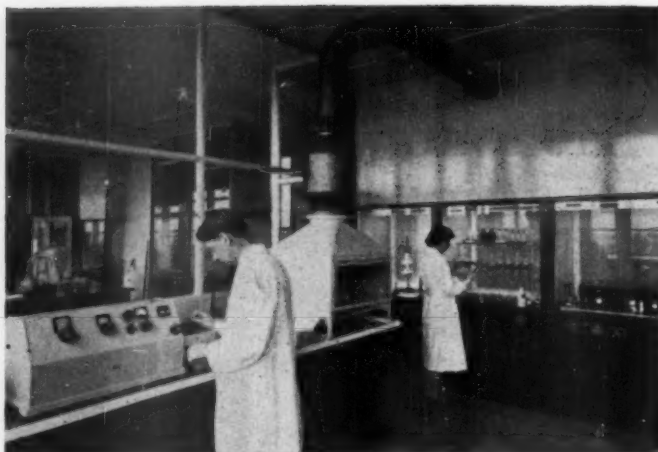
#### *Physical and Chemical Services*

The Physical and Chemical Services Division is equipped to operate a wide range of chemical, spectrographic, and X-ray analytical techniques by means of which it carries out analyses of materials for the research divisions. There is also an instrument development section which is responsible for advising on the purchase of all instruments required by the Laboratory and, where necessary, for developing new instruments. This section also provides a standardising service for all physical instruments and measurements. The glass-blowing and photographic services are also under the control of this division.

#### *Engineering Services, Intelligence and Human Sciences*

The Engineering Services Section is responsible for the construction, installation and maintenance of experimental plant required by the research divisions, the supply of power, gas, water and compressed air from Ministry of Works take-off points, and the maintenance of vehicles and portable lifting tackle. It is also responsible for the operation of the synthesis gas plant which provides the raw material for the Fischer-Tropsch work.

The Intelligence and Library Section provides the normal, editorial and library services indispensable to a research laboratory. The liaison and publicity section helps to organise exhibitions and arranges visits of parties to the Laboratory. It is also responsible for the editing and publication of reports. The library contains some 10,000 books and bound volumes of periodicals, and subscribes to more than 400 of the latter. The staff prepares a weekly bulletin, which is



**A section of the beryllium laboratory.**

circulated to all scientific staff, in which are listed the titles and authors of current papers bearing on the work of the Laboratory, together with notes of forthcoming scientific meetings and conferences, and other events of interest.

The focus on bridging the gap between bench-scale research and industrial processes by the Laboratory is reflected by the establishment of a process economics design study group, which provides a service to the Laboratory as well as to sponsors in regard to economic assessment of new projects and the evaluation of alternative lines of development for industrial application.

Towards the end of last year, the Human Sciences Section was transferred from the headquarters of the Department of Scientific and Industrial Research. Among subjects being studied by this section are work study departments, the design of keyboards and similar devices, and the supply, place and status in industry of technicians and apprentices. This section is also maintaining an index of current research on the human sciences in the United Kingdom.

#### **Mineral Processing**

Consequent upon the decision to set up the Warren Spring Laboratory, the Research Council decided that it should undertake research and sponsored work in the field of mineral processing. This decision was taken some months before the Fuel Research Station closed, and it was possible to make a little preparation during 1958. A team of chemists and engineers visited the Canadian Department of Mines and Technical Surveys, and members of the Fuel Research Station staff likely to be transferred to the new programme attended a mineral processing course at the Royal School of Mines. During the summer of 1959 four members of the staff of the Mineral Processing Section of the U.K. Atomic Energy Authority were transferred from Harwell to Warren Spring Laboratory, where an investigation of the extraction of beryllium from its ores, initiated at Harwell, is now being continued. The buildings to house the division were not completed until April, 1959, and from then until the end of September the time was spent in purchasing and installing equipment, and in recruitment and training of new staff. By the end of



1959 work had been started on sections of the basic programme and on sponsored work.

The programme of research in mineral processing has two main objectives: to effect improvements in the processes employed in the treatment of ores and minerals; and to develop methods of beneficiating hitherto unworked deposits. It includes work for the U.K.A.E.A. on a repayment basis, and the promise of further repayment work has been received from Colonial Governments and certain industrial organisations. An Overseas Mineral Processing Advisory Committee has been set up to advise on the selection and priorities of projects submitted by overseas territories. Apart from the various laboratories in which basic and applied research is carried out at bench-scale, there is a well equipped pilot-scale building containing equipment for crushing, froth flotation, wet and dry magnetic separation, separation by shaking (Wildley table), etc.

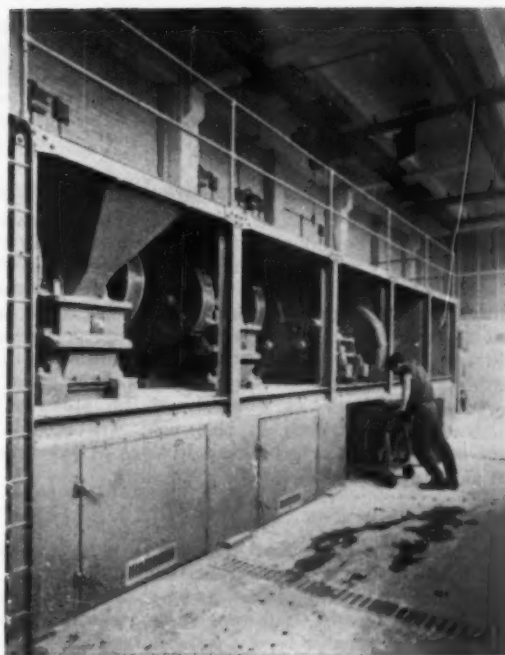
#### *Surface Chemistry of Minerals*

Attention is directed in the Report of the Director (Mr. S. H. Clarke) for 1959\* to the work on the composition of the surface of minerals and the kinetics of bubble attachment to mineral surfaces. The principal difficulty encountered in studying the surface chemistry of flotation is to obtain, in the laboratory, a reproducible surface which can be related to the surface of a mineral actually being recovered by froth flotation. This difficulty is being avoided by taking a specimen which has been subjected to the same treatment as the corresponding mineral in a typical flotation circuit and analysing surface layers of reagents adsorbed on the specimen.

Electrometric titration is being used in this study. The surface under examination is made the cathode and a platinum spiral used as the anode in a decinormal solution of an indifferent electrolyte. A high standard of cleanliness is necessary to obtain reproducible results. Experiments using spectroscopically-pure electropolished copper have given well-defined and reproducible discharge potentials for sulphide, oxide, ethyl xanthate ions, and mixtures of these three. In a typical case, the results showed that there was about 0.8 of a monolayer of xanthate on a layer, about 70 Å thick, of cuprous sulphide containing about 2% of cuprous oxide.

The ratio of the number of metallic atoms to all other inorganic atoms present in the surface of the mineral cannot be measured by the electrometric titration method but, provided the cathodic product is soluble, a large number of atomic layers can be stripped before there is any change in the potential of the polarised electrode at low current densities. This ratio does, however, determine the static potential which is acquired by an electrode when dipped in a solution containing ions common to both the solution and the surface, by reason of the electrostatic field necessary to equalise the chemical and electrostatic potentials of the ions in solution and in the surfaces.

Minerals are often treated in practice with certain inorganic reagents in order to modify the interaction between the mineral and the organic reagents used in the flotation process. The mode of action of the modifying agents is little understood, but there is good reason to believe that they might affect the surface



Plant used on an investigation of primary crushing operations on a pilot scale of up to 2½ tons per hour.

atomic composition, and thereby produce a change in the static electrode potential.

The effect on the potential of treating a number of galena specimens with a variety of sulphiding solutions has been examined, and from the observed shift in potential it is inferred that sulphiding actually brings about a decrease in the relative number of sulphide atoms in the crystal surface.

The existence of regions of different electrode potential on the mineral surface is of great practical interest, since these regions can give rise to local and inter-particle "corrosion" currents which may themselves modify the surface. This may occur either by the formation of solid corrosion products or by transference of anions and cations between the regions so as to result in the formation of a neutral uniform surface. Some fresh galena surfaces have exhibited a considerable drift of potential with time. Under these circumstances a change in adsorption characteristics dependent on the time elapsed between grinding and the addition of flotation reagents should be expected to occur. This could be important and experiments have been started to ascertain the rate of the drift and the nature of the equilibration process.

Satisfactory correlation between all the phenomena so far studied can be achieved only by removing some of the uncertainties connected with the use of naturally occurring mineral specimens. For this reason, an apparatus has been constructed for the growing of single orientated crystals by the Bridgman technique, and it is intended to repeat most of the earlier work using these crystals.

Studies of the kinetics of bubble attachment to mineral surfaces have shown that apart from the well-known uncertainties involved in the measurements and

\*"Report of the Warren Spring Laboratory 1959." Published by H.M. Stationery Office for the Department of Scientific and Industrial Research (price 3s. net).

the interpretation of the results, the actual flotation behaviour of clean artificial sulphide films did not correspond with that of the natural minerals. It is hoped that the cause of the discrepancies may be made clear as a result of the electrochemical investigations.

#### *Additive Grinding*

The first step in many beneficiation processes is the grinding of the ore to a size suitable for the separation technique to be employed. It has been stated in the literature that big savings in power consumption can be achieved by grinding in the presence of certain additives. Whether or not such a procedure is economical depends on the relative costs of power and additives. Another aspect to be considered is the possibility of complications arising at a later stage of the flotation process as a result of the presence of additives. Future work in this field will include a study of the value as grinding additives of substances used as flotation "collectors."

#### *Electrostatic Separation*

The behaviour of mineral particles in a high-tension electric field and the modification of this behaviour by various surface treatments are being studied in relation to the operation of high-tension separators. These usually consist essentially of a wire at high tension and an earthed rotating drum. It is possible to have a material stick to the drum or fall-off according to the electrical conditions and the speed of rotation of the drum, and adjustment of these conditions may lead to fairly effective separation. After a literature survey of work already carried out in this field, it was decided to study the surface conductivity and charge of particles and to examine the effect of various additives on these properties. Experience showed that measurement of the surface conductivity of the particles themselves was not reliable and work is at present in progress on the measurement of this property on ground and polished solid blocks.

#### *Hydrometallurgy*

The successful development in recent years of new extraction processes for the recovery of uranium has raised the possibility of applying these techniques to the recovery of more common metals. This will only be possible if cheaper reagents can be found to replace those used for uranium. The division has been examining the possibility of using naphthenic acids for the extraction of metals such as copper, nickel, cobalt, zinc and manganese. Commercial naphthenic acids, which are derived from crude petroleum oils and have extensive industrial uses, have not previously been considered for the recovery of metals from aqueous solutions. Their cost is only about £130 a ton, compared with the price range of £640-£1,100 a ton for the alkyl phosphoric acids used in the recovery of uranium.

With many complex and low grade ores, the wanted metals can only be extracted by leaching (extracting a soluble metallic compound from an ore by dissolving it) with water or dilute acids. It is in the treatment of these leach liquors that naphthenic acids may have a possible use. The metals which are extracted by the reagent depend on the pH of the aqueous solution of leach liquors. It has been found, for example, that copper is extracted at pH 6, whereas nickel is extracted

at pH 8. Thus, by carrying out the extraction in two stages, first at pH 6 and then at pH 8, a separation of copper from nickel can be obtained. Practical tests are now being carried out. A further factor in the use of this technique, commonly referred to as liquid-liquid extraction, is that it can be used to obtain concentrated solutions of metals from very dilute solutions. This is achieved by adjustment of the ratio of organic to aqueous phases and in fact a concentration of up to ten times can be obtained.

#### *Treatment of Slimes*

One of the difficult problems in the mineral processing field is the treatment of slimes and the division is at the moment investigating a device developed by Dr. C. R. Burch, F.R.S., of Bristol University, known as a "shaken helicoid." This is based on the "river bend" action—the fact that the upper and lower layers of the cross-section of a river move radially in opposite directions at a bend in the river's course. This motion is used, in conjunction with a vibrating motion, to separate heavier particles from lighter ones.

#### *Automation*

Contacts with the mining industry have indicated that automation may become important in mineral processing plants, and consideration is being given to a possible experimental programme. There is no doubt that with the progressively poorer ores with which the industry has to deal there is an increasing need for automatic control, as anything going to waste is of relatively greater importance than is the case with richer ores.

The larger Commonwealth countries have their own geological surveys and research establishments in this field, but the establishment of the Mineral Processing Division should go a long way towards meeting the needs of colonial and newly independent overseas territories as well as those of U.K. industrial concerns interested in this field.

### **Wiggin Alloys Renamed**

BECAUSE of the wide diversity of compositions which have been developed over the last few years to meet specific material requirements, Henry Wiggin & Co., Ltd., have announced new names for certain of their high-nickel alloys. Nimonic DS becomes Incoloy DS: this is essentially a nickel-chromium-iron alloy, useful in oxidising conditions at temperatures up to 950°C., and particularly resistant to attack from the carbonaceous atmospheres encountered in some types of bright annealing equipment and to "green-rot." Valray 1 is the new name for B.A.C. Brightray, an 80/20 nickel-chromium alloy used extensively for the coating of automobile exhaust valves. Valray can be applied either as a protective coating to the valves during manufacture, or it may be used to reclaim valves which have burnt out.

### **Wellman-Tagliaferri Agreement**

AGREEMENT has been reached between Wellman Smith Owen (Furnaces), Ltd. and Leone Tagliaferri, Milan, whereby the former company are now able to offer exclusively in the United Kingdom, electric arc melting furnaces and ferro alloy furnaces of the well-known Tagliaferri design.

# Annealing Commercially Pure Titanium

By M. A. H. Howes, M.Sc., Ph.D., A.I.M.

Metal Treating Department, Group Research Centre, Joseph Lucas, Ltd., Birmingham.

*Investigation of the annealing characteristics of two grades of commercially pure titanium has shown that for many purposes it would be satisfactory to use an annealing treatment which theoretically appears suitable for material with 25-50% deformation. As a result of this work a relationship has been established between hardness and annealed grain size.*

THE three changes which occur during the annealing of cold worked metal, namely, recovery, recrystallisation, and grain growth, are time and temperature dependent and may overlap to some extent: they are schematically represented by Fig. 1 (adapted from Sachs and Van Horn<sup>1</sup>). Burke and Turnbull<sup>2</sup> have summarised the effects occurring during annealing in seven laws, five of which may be stated as follows:

- (1) A minimum amount of deformation is necessary to cause recrystallisation.
- (2) An increase in deformation above the critical amount will cause a lowering of the temperature necessary to cause recrystallisation.
- (3) An increase in annealing time will decrease the temperature necessary to cause recrystallisation.
- (4) The final grain size will depend chiefly on the

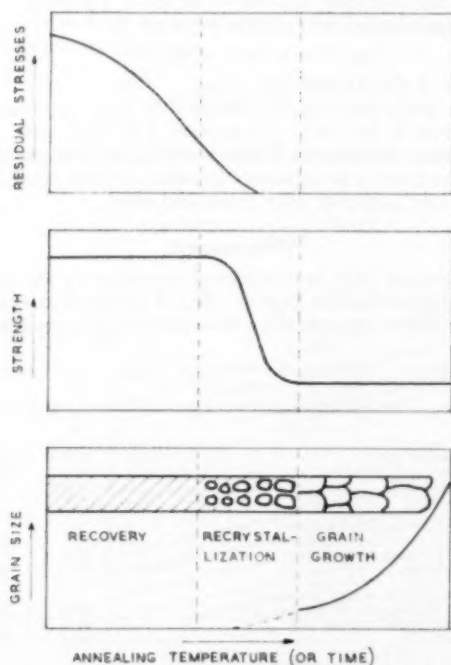


Fig. 1.—Schematic representation of recovery, recrystallisation and grain growth (adapted from Sachs).

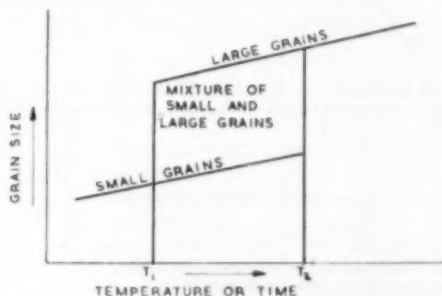


Fig. 2.—Schematic representation of discontinuous grain growth (adapted from Parker and Riisness).

degree of deformation and to a lesser extent on the annealing temperature, high degrees of deformation or low annealing temperatures giving the smallest grain size.

- (5) Continued heating after recrystallisation is complete will cause the grain size to increase.

From the practical point of view the important conditions for interstage annealing are the time and temperature necessary to complete the recrystallisation stage. The ideal conditions would produce a uniform grain size, free from stress and at the minimum hardness. Law 4 indicates that this would be a difficult matter with a range of deformation such as would be present in a drawn article. It may be possible to produce a reasonably uniform grain size, however, by choosing conditions which ensure that by the time recrystallisation is complete in the least severely worked region, then grain growth to an approximately equal size has occurred in the severely worked zones which have recrystallised earlier.

Grain growth should be avoided in regions subjected to low stresses, since it may proceed in a discontinuous manner difficult to control. A schematic representation of discontinuous grain growth after Parker and Riisness<sup>3</sup> is given in Fig. 2.

## Experimental Work

### Materials

Two types of commercially pure titanium were used for the annealing tests. Material A was of British manufacture (I.C.I. 120) and B was of earlier American origin (T.I. 75A): the composition of the two materials is shown in Table I, and the physical properties are set out in Table II. Table II shows that the main effect of the impurities in sample B is to increase strength and



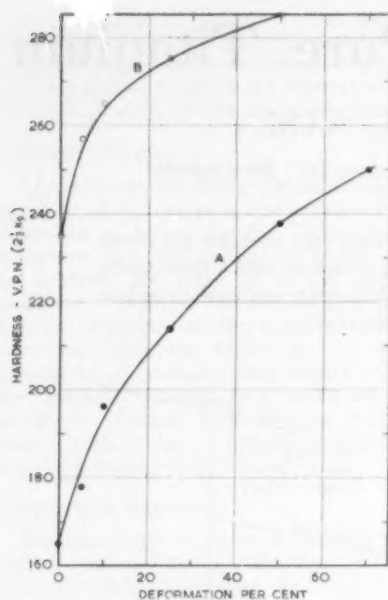


Fig. 3.—The hardness of commercially pure titanium after deformation.

hardness and reduce ductility. This material also shows directional properties.

#### Cold Working of Material

Material A was divided into five batches and deformed by cold rolling 5, 10, 25, 50 and 70%. Material B was deformed 5, 10, 25 and 50%. The mill available was not capable of reducing material B to the 70% stage.

#### Heat Treatment of Specimens

The specimen size used was  $\frac{3}{4} \times \frac{1}{4}$  in., this being the most convenient for handling and metallographic examination. A range of specimens was treated at temperatures of 300, 400, 500, 550, 575, 600, 625, 650 and 700° C. for 5, 15 and 30 minutes and 1, 3, 8 and 23 hours. The specimens were treated in a forced air tempering furnace which could be controlled within  $\pm 2^\circ$  C. An allowance of 30 seconds was made to enable the specimens to reach furnace temperature.

#### Examination of Specimens

The specimens were prepared for metallographic examination and etched in a mixture of 1% hydrofluoric acid and 6% nitric acid for grain size measurement. The hardness measurements were made on the same specimen using a diamond pyramid machine and a load of 2½ kg. The specimens were examined and tested away from the edge to avoid the contamination which occurs when titanium is heated in air.

TABLE I.—COMPOSITION OF COMMERCIAL PURE TITANIUM

Material	C%	Fe%	N%	O%	H%
A	0.05	0.10	0.03	0.02	0.009
B	0.10	0.10	0.026	0.16	0.024

Material B generally contains far more impurities, especially oxygen, than material A.

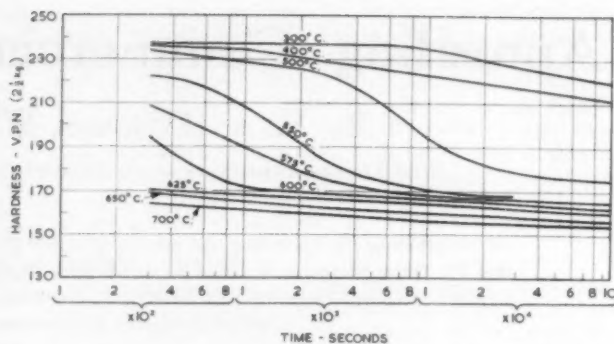


Fig. 4.—Hardness of material A after 50% deformation and heat treatment at various temperatures.

### Results

The hardness of the titanium after cold working, shown in Fig. 3, indicates that the hardening effect is quite marked, especially during the first 25% deformation. The results of hardness tests on the heat treated material were recorded in the form shown by Fig. 4, which gives the hardness of material A with 50% deformation followed by treatment at different temperatures for the full range of time.

Beck and co-workers<sup>4</sup> have shown that grain growth continues with time at any one temperature until the average grain size approaches the thickness of the specimen used. They found that grain size  $D$  could be related with total annealing time  $t$  by the equation

$$D = Kt^n \quad (1)$$

where  $K$  and  $n$  are constant at constant temperature. There may be small deviations at short annealing times. The logarithmic form of this equation is

$$\log D = n \log t + \log K \quad (2)$$

This is of the straight line type.

The grain size results obtained in this investigation are given in the form of equation 2 in Figs. 5-8. The grain size of material B deformed to 25% or greater is not given, since no apparent growth occurred during the maximum period of heat treatment used.

### Discussion

Annealing may be considered complete at the end of the recrystallisation period. Fig. 1 shows that the end of this period is marked by the start of grain growth and

TABLE II.—PHYSICAL PROPERTIES OF COMMERCIAL PURE TITANIUM

Property	A	B
Sheet Thickness (in.)	0.048	0.050
Hardness (V.P.N.—2½ kg.)	164	235
Grain Size (mm.)	0.035	0.037
Erickson (mm.)	9.9	6.3
<i>Properties at 0° to Rolling Direction:</i>		
Ultimate Tensile Strength (tons/sq. in.)	27.8	43.2
0.1% Proof Stress (tons/sq. in.)	23.2	37.7
Elongation (%)	29.0	24.0
<i>Properties at 45° to Rolling Direction:</i>		
Ultimate Tensile Strength (tons/sq. in.)	28.6	41.4
0.1% Proof Stress (tons/sq. in.)	23.4	35.8
Elongation (%)	29.0	22.0
<i>Properties at 90° to Rolling Direction:</i>		
Ultimate Tensile Strength (tons/sq. in.)	29.0	
0.1% Proof Stress (tons/sq. in.)	23.0	
Elongation (%)	30.0	



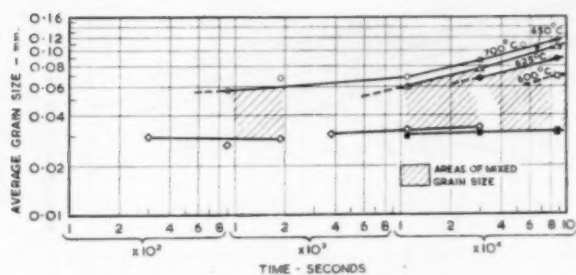


Fig. 5.—Grain growth of material A deformed 5% and heat treated at various temperatures.

by the attainment of the minimum hardness. Both these factors have been used in an attempt to determine the minimum annealing conditions for commercially pure titanium.

It has already been noted that the grain size after recrystallisation is dependent on the amount of deformation. This effect is illustrated by Fig. 9, which shows the grain size for materials A and B after deformation and recrystallisation. These grain sizes were estimated for each amount of deformation by determining the smallest average grain size showing a recrystallised structure free from evidence of stress. If the grain growth curves given in Figs. 5-8 are produced back to the values indicated by Fig. 9, the approximate time at which grain growth began may be determined. This procedure is slightly more complicated in the case of material A subjected to 5% deformation, since the resultant grain growth is of the discontinuous type illustrated in Fig. 2, but a fair estimate may be made.

It was shown by Fullman<sup>5</sup> that the general expression of transformation kinetics

$$\frac{dU}{dt} = f_0(U) e^{-Q/RT} \quad (3)$$

may be integrated and rearranged in terms of logarithms to the base 10

$$\log [f_1(U) - f_1(U_0)] = \log t - \frac{Q}{2.3RT} = \log t - \frac{C_1}{T} \quad (4)$$

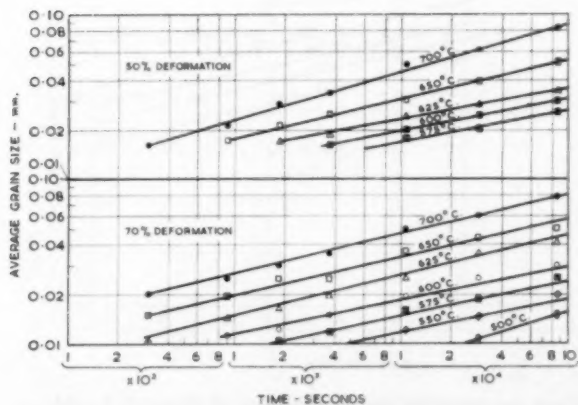


Fig. 7.—Grain growth of material A deformed 50% and 70% and heat treated at various temperatures.

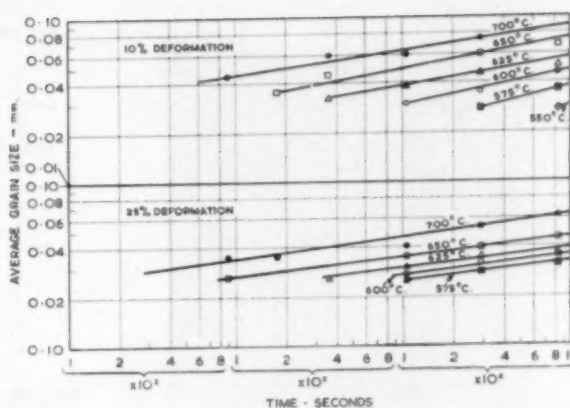


Fig. 6.—Grain growth of material A deformed 10% and 25% and heat treated at various temperatures.

where  $U$  = fraction transformed

$f_0(U)$  = a function of  $U$

$T$  = absolute temperature

$R$  = gas constant

$Q$  = activation energy of process, which may be considered constant within narrow limits

$t$  = time

$C_1$  = constant

The left hand side of equation 4 is a function of the extent of transformation, and may be taken as constant if a particular fraction of transformation is considered. Thus,

$$C_2 = \log t - \frac{C_1}{T}$$

where  $C_2$  is a constant, or

$$\log t = \frac{C_1}{T} + C_2 \quad (5)$$

which is of straight line form.

If the grain size information is plotted in this form the straight line relationship is seen to exist (Fig. 10).

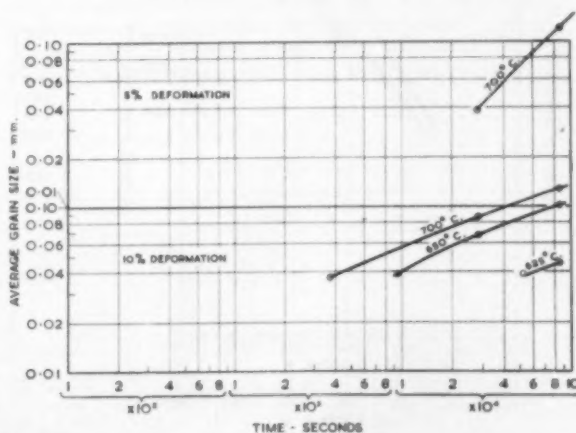


Fig. 8.—Grain growth of material B deformed 5% and 10% and heat treated at various temperatures.

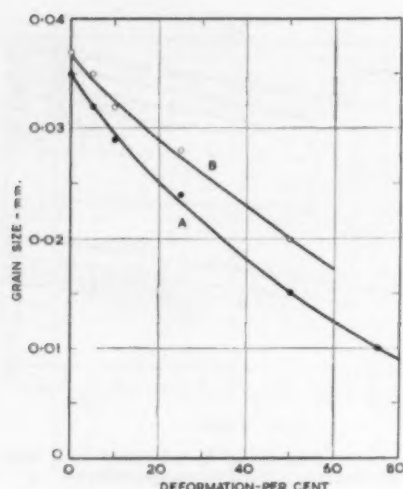


Fig. 9.—Grain size of deformed titanium after recrystallisation.

A similar diagram could not be produced for material B since insufficient grain growth information was available. Burke and Turnbull<sup>3</sup> have suggested that a highly dispersed second phase may inhibit grain growth very effectively, which leaves little doubt that the extra impurities in material B are forming a phase which has this effect.

The second method available for the determination of the end of the recrystallisation period depends on the approximation that minimum hardness is expected at the end of this period. This is not strictly true, since the hardness will continue to fall slightly due to grain growth.

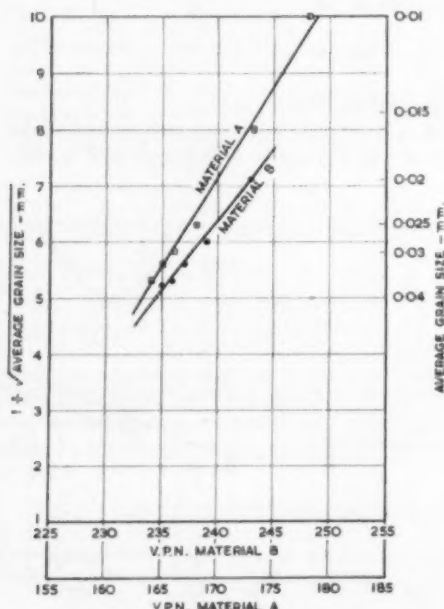


Fig. 11.—The relationship between grain size and hardness of annealed titanium.

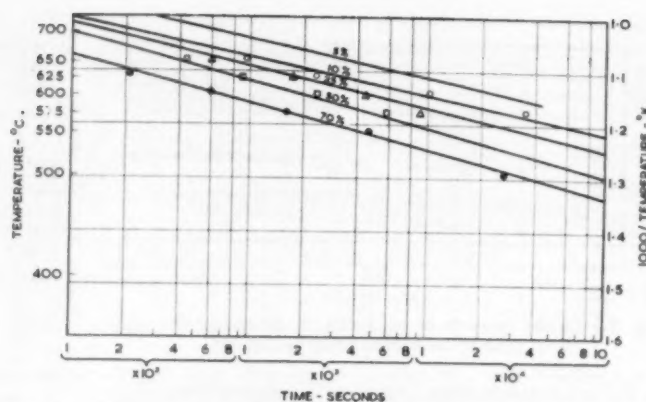


Fig. 10.—Time for complete recrystallisation of material A cold worked by various degrees of deformation (based on grain size measurements).

It is therefore necessary to know the hardness related to the grain size produced at the end of the recrystallisation period.

Petch<sup>6,7</sup> and Stroh<sup>8</sup> have related fracture stress  $S$  and lower yield point with grain size  $D$  by equations which may be converted into the form.

$$S = C_3 + C_4 D^{-1/2} \quad (6)$$

where  $C_3$  and  $C_4$  are constants. Since fracture stress is approximately proportional to hardness.

$$\text{Hardness} = C_5 + C_6 D^{-1/2} \quad (7)$$

where  $C_5$  and  $C_6$  are constants. This is of straight line form.

This relationship is shown to be correct by Fig. 11, which enables the hardness of the recrystallised material to be estimated. Using the hardness results for materials A and B, Figs. 12 and 13 were constructed similar to Fig. 10. Comparison of Figs. 10 and 12 for material A shows that there is good agreement up to 25% deformation, but the more highly worked material apparently reaches the annealed hardness before the grains have completely recrystallised. It is possible that some grain growth restriction is occurring, thus making the experimental observation of the start of grain growth difficult.

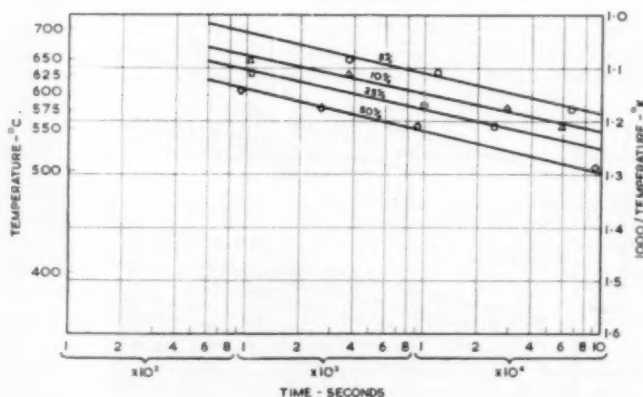


Fig. 12.—Time for complete recrystallisation of material A cold worked by various degrees of deformation (based on hardness measurements).

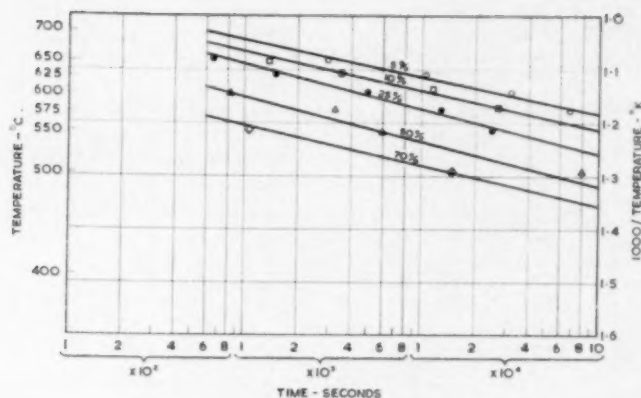


Fig. 13.—Time for complete recrystallisation of material B cold worked by various degrees of deformation (based on hardness measurements).

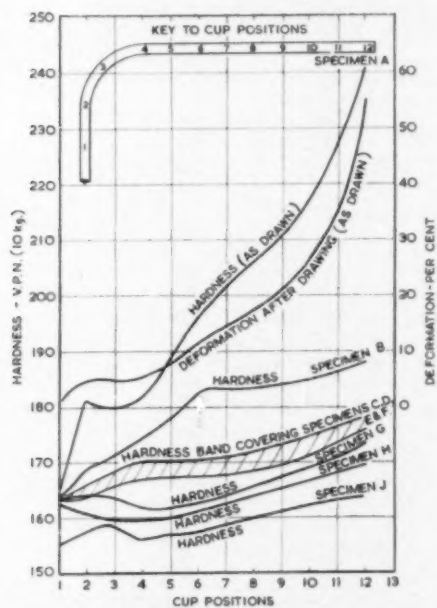


Fig. 15.—Hardness plots along heat treated titanium cup sections.



Fig. 14.—Cup drawn from a 4.900 in. diameter blank of material A.

The curves, based on hardness measurements, given in Figs. 12 and 13 are likely to be the most accurate and are preferred for experimental use.

#### Experimental Confirmation of Results

A 4.900 in. diameter blank of material A was drawn into a cup of 2.00 in. internal diameter (Fig. 14). This would be expected to give a range of deformation from maximum at the open end of the cup to nearly zero deformation at the bottom of the cup. The cup was then sectioned into sectors and the sectors were heat treated at temperatures between 575° C. and 650° C. for 15 minutes and 3 hours.

Subsequently, grain size estimations and hardness plots were made at twelve positions along the section and all these details are given in Table III and Fig. 15. Table III shows the grain size before and after the treatments. The hardness plots are given in Fig. 15 together with a deformation plot estimated from the as-drawn hardness (Specimen A).

The results in Table III show that recrystallisation may be considered complete after treatments of 650° C. for 15 minutes or 625° C. for 3 hours. The parts of the cup deformed greater than 25% are free from evidence of work after 15 minutes at 600° C. or 3 hours at 575° C., while parts deformed greater than 10% appear free after

TABLE III.—HEAT TREATMENT AND GRAIN SIZE OF CUP SECTORS DRAWN FROM MATERIAL A

Specimen Letter	Heat Treatment after Drawing		Average Grain Size (mm.) at Cup Position Indicated on Fig. 15											
	Temperature (°C.)	Time (hr.)	1	2	3	4	5	6	7	8	9	10	11	12
A .. .. .	—	—	0.035	D	D	D	D	D	D	D	D	D	D	D
B .. .. .	575	15 min	0.035	D	D	D	D	D	D	D	D	D	0.007	0.005
C .. .. .	575	3 hr	0.035	D	D	D	D	D	D	D	0.015	0.012	0.010	0.010
D .. .. .	600	15 min	0.035	D	D	D	D	D	D	D	0.015	0.012	0.012	0.010
E .. .. .	600	3 hr	0.035	D	D	D	D	0.020	0.020	0.017	0.015	0.015	0.012	0.012
F .. .. .	625	15 min	0.035	D	D	D	D	0.025	0.025	0.020	0.017	0.015	0.012	0.010
G .. .. .	625	3 hr	0.035	0.035	0.045	0.050	0.045	0.035	0.025	0.020	0.020	0.017	0.012	0.015
H .. .. .	650	15 min	0.035	0.035	0.037	0.037	0.040	0.030	0.027	0.027	0.027	0.025	0.025	0.020
J .. .. .	650	3 hr	0.035	0.035	0.035	0.040	0.060	0.050	0.045	0.045	0.045	0.045	0.045	0.045

D—Indicates evidence of deformation.

15 minutes at 625°C. or 3 hours at 600°C. The information given by Fig. 12 is in general agreement with the above experimental results but tends to be pessimistic at the shorter times.

The hardness results from Fig. 15 show that recrystallisation would be far enough advanced after periods of 15 minutes at 600°C. or 3 hours at 575°C. for the material to be soft enough for further working in most instances. These periods correspond to those given by theoretically complete recrystallisation of material deformed 25-50%. There is evidence that discontinuous grain growth occurs during treatment at 650°C. and this temperature should be avoided.

### Conclusions

- (1) The annealing characteristics of two grades of commercially pure titanium have been examined and curves have been drawn showing the relationship between temperature, time, recrystallisation and deformation. There appears to be no significant

difference in annealing characteristics between the two grades. These curves are in general agreement with practical results but tend to be slightly pessimistic at the shorter times of treatment.

- (2) The practical results show that for many purposes it would be satisfactory to use an annealing treatment which theoretically appears suitable for material with 25-50% deformation.
- (3) A relationship has been established between hardness and annealed grain size (equation 7).

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## Prespheroidising of High Speed Steel\* Change of Cast Structure by Heat Treatment Alone

**H**IGH speed steel is the most popular material for the manufacture of cutting tools. Its unique properties are conferred by the presence of substantial quantities of alloying elements, notably tungsten. Some typical compositions are set out in Table I.

Because of the particular composition of high speed steel, the freezing process is extremely complex and gives a structure like that shown in Fig. 1. There are large colonies of eutectic, and the structure is by no means homogeneous. This structure is normally not acceptable for tools, and has to be broken down by hot work: forging, rolling or extrusion. The progress of this break-up is shown in Fig. 2 (billet stage) and Fig. 3 (finished bar): the eutectic colonies of Fig. 1 are elongated into stringers of carbide, but the structure remains heterogeneous. The presence of gross eutectic colonies can cause the steel to break up during hot work. The stringers are generally considered to be undesirable: they can cause distortion or failure during heat treatment, and have been blamed for countless failures of high speed steel tools in service.

Until recently it was believed that heat treatment alone could not alter the cast structure, Fig. 1. However, work in the laboratories of the British Iron and Steel Research Association has shown that it can. The irregularities in Fig. 1 are thought to be caused by lack of equilibrium; that is, in the freezing operation there was insufficient time for completion of a peritectic reaction. If this reaction were allowed to proceed, it should, it was reasoned, give a structure free from eutectic, and should therefore eliminate the coarse stringers shown in Fig. 3.

The technique adopted is to re-heat the as-cast steel to the reaction temperature (about 1,320°C.), at which

TABLE I.—COMPOSITION OF FOUR TYPES OF HIGH SPEED STEEL

Type	C%	W%	Cr%	V%	Mo%	Co%
18-4-1	0.7	18.0	4.0	1.0	—	—
6-5-2	0.85	6.0	4.0	2.0	5.0	—
18-4-1+Co	0.75	18.0	4.5	1.25	—	5.0
9-3-2-8	1.33	9.0	4.25	2.5	2.2	8.5

the eutectic actually becomes molten. But the steel is then in the same state as it was just before the ingot froze, and by holding it at this temperature reaction takes place between liquid and solid to give a new solid phase without eutectic. This operation has been termed prespheroidising, because it enables the carbide (eutectic) to be spheroidised, or broken up into small globules, before any hot work is carried out.

The effect of prespheroidising on an ingot structure is shown in Fig. 4 (compare Fig. 1, untreated). After hot working the structure becomes comparatively uniform (compare Figs. 5 and 6 with Figs. 2 and 3) with an almost complete absence of stringers.

It must be emphasized that the process is not yet a commercial proposition. The practical difficulties involved in the treatment of large masses of metal at the temperatures involved are great. An accidental temperature rise of as little as 5°C. could be disastrous, and the metal might melt sufficiently to lose its shape and be completely ruined. Also it has yet to be established that the treatment improves the properties of high speed steel, and if it does whether the improvement is sufficient to warrant the expense involved. The carbide particles in prespheroidised samples are larger than is usually considered desirable, and this may have an embrittling effect.

There are two applications, however, where prespheroidising could be of immediate value. A small proportion of high speed steel tools are precision-cast to shape, and are heat treated without any hot work being carried out. These tools contain eutectic, but, because of the small size of the section, the structure is generally finer than in ingots, Fig. 7, where the magnification is

\*Based on B.I.S.R.A. reports MG/L/197/58, "The Reduction of Carbide Heterogeneity in High-Speed Steel by Thermal Treatment of the Ingot," by G. Hoyle and E. Ineson; MG/L/44/59, "An Investigation of the Prespheroidising Process by Thermal Analysis," by A. Hollingsworth and G. Hoyle; and MG/L/50/59, "The Reduction of Carbide Heterogeneity in High Speed Steel by Thermal Treatment of the Ingot: Further Experimental Results," by D. B. Hammond and G. Hoyle. These reports, and further copies of this summary, can be obtained from the Information Officer, B.I.S.R.A., 11 Park Lane, London, W.1.



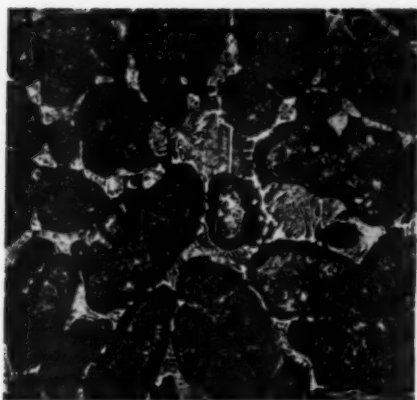


Fig. 1.—High-speed steel ingot—as cast

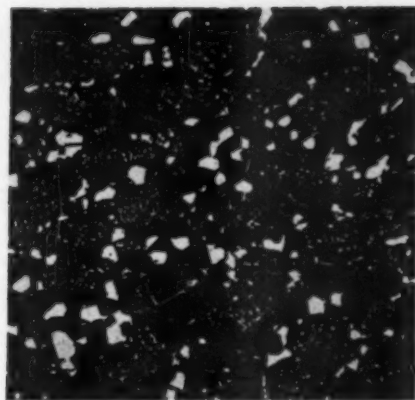


Fig. 4.—High-speed steel ingot—  
prespheroidised

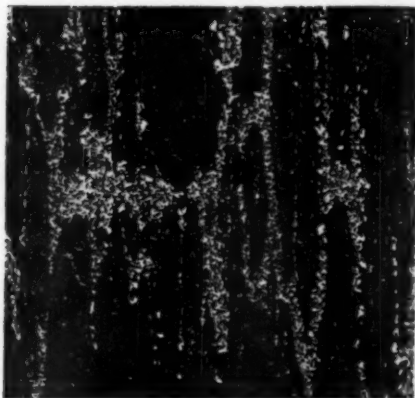


Fig. 2.—Two-inch billet forged from ingot

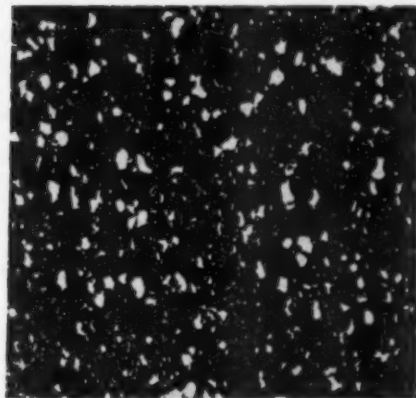


Fig. 5.—Two-inch billet forged from  
prespheroidised ingot

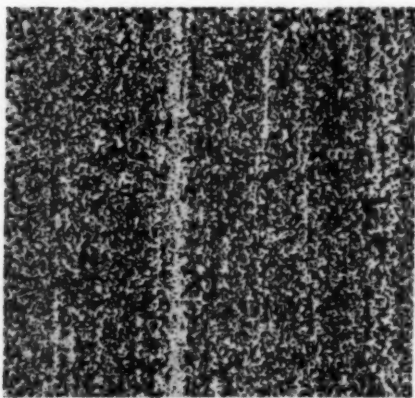


Fig. 3.—Half-inch bar rolled from ingot

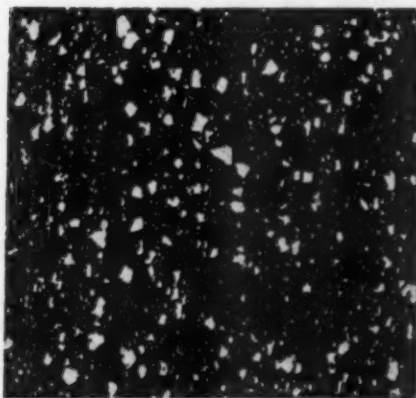


Fig. 6.—Half-inch bar rolled from  
prespheroidised ingot

Figs. 1-6.—Photomicrographs showing the effect of prespheroidising on the structure of high speed steel in the ingot, billet and bar forms.

× 100

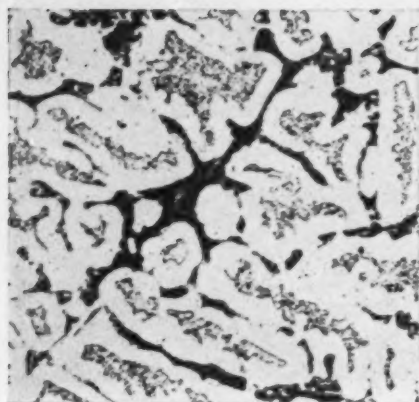


Fig. 7.—Cast cutter—untreated

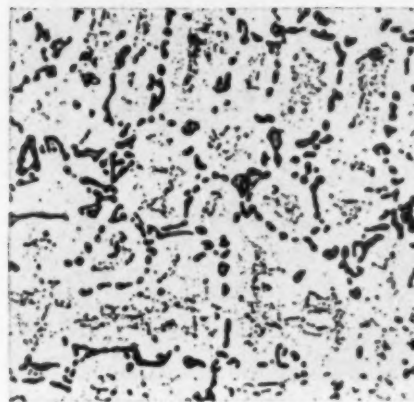


Fig. 8.—Cast cutter—prespheroidised

Figs. 7-8.—Photomicrographs showing the effect of prespheroidising on the structure of precision cast high speed steel cutters.

× 500

×500—five times as great as that in Fig. 1. The prespheroidising temperature is only about 30° C. above the normal hardening temperature for high speed steel, so that in this case the tool can be prespheroidised, quenched, and tempered, and is then ready for final grinding and use. The masses involved are small, and this means that temperature control is much easier—either using a muffle furnace or, preferably, a salt bath.

Handling is also much simpler. Fig. 8 shows the prespheroidised structure.

A further application is the restoration of overheated tools. If the correct hardening temperature is exceeded, a steel with the structure shown in Fig. 3 will revert to a eutectic structure similar to that shown in Fig. 1. Such tools can be prespheroidised in the same way as cast cutters and an expensive rejection may be avoided.

## Welding Industry Development in Russia

THE British Welding Research Association has now completed the cover-to-cover translation and publication in English of the Russian monthly welding journals *Avtomaticheskaya Svarka* and *Svarochnoe Proizvodstvo* for 1959. Some novel and surprising techniques have been disclosed, in part brought about by the remoteness of some of the areas in Russia where industry must rely on indigenous supplies of raw materials, and by the enormous distances which have to be covered in such programmes as rail, pipe and electrical transmission line construction.

Thus the development of flat coiled and welded aluminium pipeline was described in the January 1959 issue of *Avtomaticheskaya Svarka*. On site the coils are unrolled and restored by compressed air to give continuous runs up to 600 m. long. Again, in the October issue of the same journal, the use of water vapour as a shielding atmosphere for arc welding is described—a matter of expediency where the cost and time involved in transporting conventional shielding gases would be prohibitive.

With capital equipment at a premium and heavily worked, great emphasis has been placed on repair, rebuilding and hard facing by welding, using "fire cracker" techniques with horizontal flat and "powder tape" electrodes. Also considerable attention is being paid to automation and to mechanisation, not only for internal expansion but as a means of intensifying competition with Europe and the West.

Another aspect which is particularly evident is the enthusiasm and earnestness of the Russian engineer,

welder and technician in submitting ideas and in giving full vent to criticisms both of other authors and of the journals themselves. Some of the information published is outdated by Western standards—it may even have been proved erroneous, but values must be based on overall technical and technological developments, and even these articles have a distinct value as a stimulus to criticism and counteraction. The important fact is that there is a marked awareness by the Russians of their technical shortcomings and that there is a powerful drive to overcome these, with—in many cases—world-shaking results!

Since publication of English translations began there has been a continuously increasing call for them from many parts of the world, including the U.S., Japan, the Commonwealth and Western Europe. Results from the U.K., while showing some increase, have been slow in comparison.

## Firth Company Rebuilding

IN rebuilding the fire damaged wet drawing department and copper coating section at the Warrington works of the Firth Co., Ltd., the most modern methods of production and handling have been incorporated in the new installation. These include drum packing machinery and conveyor feed from the finishing blocks to the testing and weighing-off department. The latest stripping and crane arrangements have been installed in the copper coated steel section and a completely new type of plastic acid resisting floor has been laid down. The whole project will substantially increase the output in fine sizes of wire both bright and galvanised and all classes of wet drawn and copper coated steel wire.

# Casting Variables Affecting the Machinability of Grey Iron

By E. A. Loria \*

*The machinability of cast iron is related to the microstructure, rather than to the usual criteria of hardness and strength. The influence of chemical factors on microstructure has been explained chiefly on the basis of the tendency of each constituent element to accelerate or inhibit graphite formation during solidification. The purpose of this article is to discuss the influence on machinability of such physical factors as pouring temperature, rate of cooling, mould conditions and inoculation practice.*

IN the machining of cast irons, it has been shown that the cutting characteristics are related to the microstructure of the iron rather than to the usual criteria of hardness and strength.<sup>1,2</sup> In different types of machining such as turning, drilling, reaming and milling, different iron microstructures may stand in different orders, but basically the constituents which produce abrasive wear on the cutting tool lower machinability. While the casting skin, inclusions and sub-surface discontinuities, as well as other extraneous causes, are detrimental to machinability, the distribution, size and type of graphite and free carbide have the most important bearing on how the machine operator classifies the casting. The writer has conducted a number of machinability testing procedures and all are predicated on this concept. These have included lathe turning tests on rings and bars, measuring tool wear;<sup>3,4</sup> constant-pressure lathe turning tests on bars;<sup>5,6</sup> drill penetration tests under constant load and speed on bars and castings;<sup>7</sup> and lathe turning tests on rings, measuring cutting temperature and force.<sup>8</sup> Actually a combination of such test methods is needed to provide a complete evaluation of the machinability of a particular iron composition.

Several factors are known to influence the microstructure of cast iron. Generally these factors can be grouped into two broad classifications: chemical factors, which are related to melt composition; and physical factors, which include thermal and "hereditary" effects. The iron-carbon-silicon eutectic may solidify from the liquid state in one of two forms under commercial rates of cooling: either as austenite and carbide or as austenite and graphite. At room temperature there will be pearlite and carbide or pearlite and graphite, respectively. In the absence of alloying elements, the mode of solidification into one or the other pair or into a mixture of the two, is determined by (a) the silicon content, and (b) the rate of cooling from the molten state. In general, the influence of chemical factors has been explained chiefly on the basis of the tendency of each chemical element in the melt to accelerate or inhibit the formation of graphite during solidification. The influence of most physical factors is still relatively unexplained. Some of these would include pouring temperature, rate of cooling, mould conditions, and inoculation practice. The purpose of this article is to discuss the influence of these factors on machinability.

## Thermal Factors

In practice, the minimum thickness of section in which any given class of grey iron may be poured is more likely to depend on the cooling rate of the section than on the fluidity of the metal. The most important effect of mass in casting grey iron is on the graphite size and distribution, and the amount of carbon in the combined form. These, in turn, are the two most important constituents controlling machinability. For any given grey iron composition, the rate of cooling from the freezing temperature to below 1,200° F. (650° C.) determines the ratio of combined carbon to graphitic carbon, which controls the hardness and strength of the iron. For this reason, the effect of section is considerably greater than in the more homogeneous ferrous metals where cooling rate does not affect the form of the carbon content on a macroscopic scale. The most appropriate measures of the section sensitivity of any given iron composition are the chill and wedge tests which serve as indicators of the general properties of the iron when cast in light sections. Therefore, the relation between chilling tendency and machinability can be estimated for each particular melting practice. Brinell hardness and strength are related to the total carbon content + one-third the silicon content, but chill depth is related more closely to total carbon content + one-and-a-half times the silicon content.<sup>9</sup> Therefore, there cannot be a direct relationship between hardness and chilling tendency.

In dealing with chilling properties, it is a fact that there may be a marked difference in the response of any particular iron to a forced chill as compared with a natural chill. The point at which free carbide begins to form in a sand-cooled casting will not necessarily bear more than a rough relationship to the depth to which the chill will penetrate when the rate of cooling is markedly increased by the provision of metallic chills or by the extension of the depth of chill in a sand-cooled wedge. Although experience has shown that such control tests can only be partially related to chemical analysis, the latter is really only controlled as a means of getting reproducible mechanical properties in commercial castings. It is well known that these properties can vary widely for the same analysis when, for example, charging or melting practice is varied. It appears that the standard control test, whether by forced chill or by sand-cooled wedge, will give a better index of the machinability of the iron than the chemical analysis. Nevertheless, both procedures are needed for control purposes and their use is completely justified,

\* At the time of this investigation, the author was at Mellon Institute of Industrial Research, Pittsburgh and The Carborundum Company, Niagara Falls. Now, metallurgical development manager, Climax Molybdenum Company, Pittsburgh.



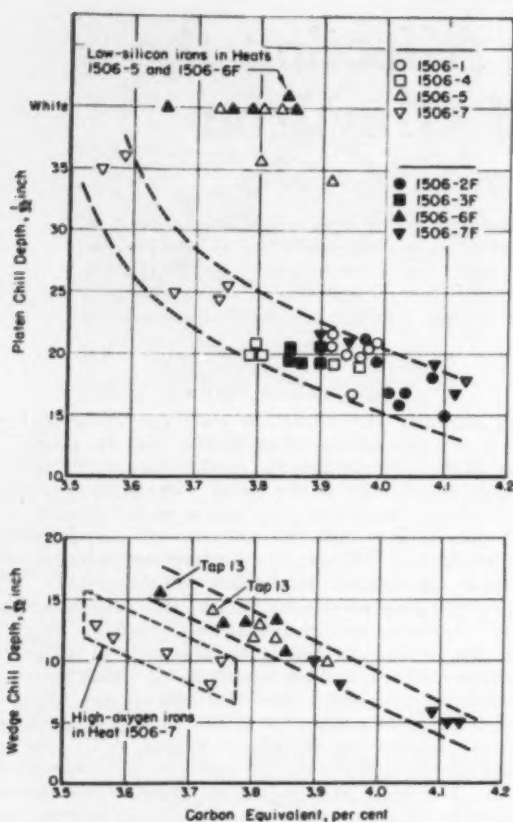


Fig. 1.—Relation of chill depth to carbon equivalent for irons melted in small cupola under normal and oxidising practice. Oxygen values corresponding to heat symbols and carbon equivalents will be found in references 10 and 11 listed at the end of the article.

even though the results of the one may not agree entirely with the results of the other.

Pouring temperature has an important influence on the chill behaviour of cast iron. To determine its exact influence, one must bear in mind that any set of results pertain only to the conditions of the particular experiment. As an example, in a statistical study of forced chill and wedge tests poured from automotive grade iron which had a carbon/silicon ratio of 1.5 to 2.0, the forced chill depth increased as the temperature was lowered from 2,850°–2,725° F. (1,565°–1,495° C.). When these same irons were held in the ladle and

poured in the lower range of 2,250°–2,450° F. (1,230°–1,345° C.), the data showed that there was no influence of pouring temperature on chilling tendency in sand-cooled wedges. For both the forced and sand-cooled wedge tests, the irons with the lowest carbon/silicon ratio had the lowest chill depths throughout the temperature range, which suggests the important effect of silicon in controlling chill. On the other hand, for roll-type cast iron which had a high carbon/silicon ratio of 4.0, the forced chill depth decreased as the pouring temperature was lowered to 2,450° F. (1,345° C.), this being particularly true in melts superheated to 2,900° F. (1,595° C.).

The important thing for the engineer to understand is that the cooling in the mould at the time of freezing is the controlling variable in the possible formation of chill in light sections. If a thin section is in the drag or near the gate, the flow of hot metal heats the mould and thereby decreases the rate of cooling. An example of the importance of pouring temperature in such a casting design is illustrated in Table I. The difference in tool wear machinability between each pair of base and cylinder irons is attributed to the difference in pouring temperature. Pouring relatively cold iron into this critical section precludes such a result. If one considers the thermal factors involved, it is expected that the cooling rate during solidification of a metal poured at low temperatures into a mould should be greater than that of metal poured at a higher temperature into a similar mould. This conclusion stems from the assumption that the higher the temperature of the metal entering the mould, the more the mould will be heated prior to solidification of the metal with consequent reduction in cooling rate during solidification.

There are instances where the increase in carbide stability cannot be ascribed to variation in pouring temperature or the content of the normal elements in cast iron. In these cases, the chilling properties are frequently attributed to what has been termed "oxidised iron." A fairly recent systematic study of the metallurgical quality of nine heats of automotive grade iron, melted in the Battelle 10 in. diameter cupola under normal and oxidising practice, gave the most complete correlation of vacuum fusion oxygen analysis of the metal, cupola slag analysis and metal chemistry to be found in the literature.<sup>10,11</sup> This study produced irons ranging from 0.0012 to 0.0068% oxygen which were poured into a variety of test bars.

A statistical study of the chill test results shown in Fig. 1, reveals that irons containing 0.0022–0.0046% oxygen had forced chill depths that were  $\frac{1}{16}$  in. below the expected value for their composition, and irons containing less than 0.0016% oxygen had forced chill

TABLE I—TOOL LIFE TESTS ON GREY IRON RINGS

Type of Iron	Composition (%)							Pouring Temperature		Average Number of Ring Faces Machined†	Volume of Metal Removed (cu. in.)‡
	T.C.*	Si	Mn	S	Cu	Cr	C.E.†	(°F.)	(°C.)		
Base iron	3.40	2.18	0.72	0.10			4.12	2,550–2,510	1,400–1,375	38	59.0
	3.39	2.12	0.71	0.10			4.09	2,650–2,600	1,455–1,425	50	78.5
Cylinder iron	3.35	2.33	0.69	0.10	0.80	0.28	4.12	2,550–2,500	1,400–1,370	15	25.5
	3.50	2.26	0.73	0.10	0.80	0.31	4.25	2,620–2,590	1,440–1,420	20	31.4

\*—Total carbon.

†—Carbon equivalent.

‡—As-cast surface machined to a depth of  $\frac{1}{16}$  in. on 9 in. O.D. × 7 in. I.D. ×  $\frac{1}{4}$  in. thick rings individually cast in core sand.

§—Based on cubic inches of metal removed for 0.020 in. wear land at 300 ft./min. cutting speed and a feed of 0.009 in./rev. Grade 44A carbide tools used with a nose radius of 0.030 in. Tool angles were: back rake 0°, side cutting edge 0°, side relief 6°, side rake 4°, and cutting edge 8°, and relief 6°.



depths averaging  $\frac{1}{8}$  in. greater than the expected chill depth. This shows that higher oxygen contents, within the range of 0.0012-0.0068%, did not increase forced chill depth. With the wedge tests, there was a good linear relationship between carbon equivalent of the irons and their chill depths. The results indicated that oxygen contents below 0.005% in the iron did not affect chill depth. In the 0.005-0.007% range, oxygen appeared to have a slight graphitising tendency. These results are in disagreement with the common belief in grey iron foundries that "oxidised irons" produce high chill depths. However, common use of the word "oxidised" to describe certain irons sometimes encountered in the foundry may be completely erroneous.

### Mould Conditions

Even under ideal thermal and chemical factors, one encounters variations in machining the as-cast surface of castings. The outer surface of a casting usually has a different structure, which is less easily machined than the underlying metal. This is particularly true in thin section castings which solidify at a more rapid rate. A good example is set forth in Table II, which presents the results on machining the parallel faces of the  $\frac{1}{8}$  in. thick rings of cylinder engine block analysis. For each batch, twenty-five test rings were cast in core sand at a time, the metal being taken from a 2 ton ladle. The irons were made in recuperative hot-blast cupolas lined to 76 in. and front-slugging. The metallic charge was 5,000 lb. and the melting rate was around 30 tons/hr.

As for all tool wear machining tests reported herein, a modern lathe having a variable speed drive was used in facing the rings. The  $\frac{1}{8}$  in. depth of cut was made with grade 44A carbide tools of the geometry given in Table I. At the 300 ft./min. speed, each machining test was repeated or rechecked, and thereby an average value of the number of ring faces machined for 0.020 in. wear land was obtained. The results listed in Table II verify that the as-cast surface is much more difficult

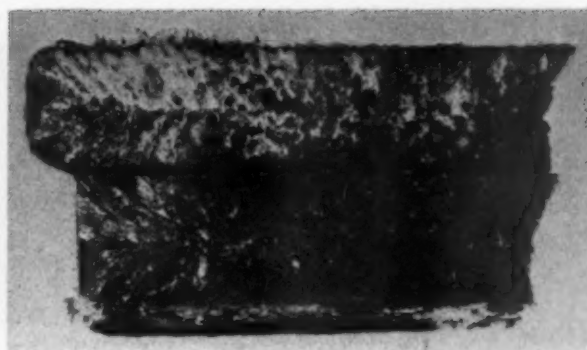


Fig. 2.—Macrograph of  $\frac{1}{8}$  in. thick step bar cross section of 3.30% T.C., 1.87% Si iron cast in wet sand (top) and dry sand (bottom). Etched in picral.  $\times 3$

to machine than the underlying metal. The tool life on making facing cuts on a pre-machined surface is 2-4 times that on the as-cast surface of this critical section. Batch B and F rings which were cast to a lower temperature range had lower machinability at both levels, which again points to the importance of temperature in pouring iron into this particular mould design.

Vivid examples of the detrimental effect of mould moisture on the casting of thin sections of grey iron step bars are shown in Figs. 2 and 3. Casting the same iron in wet vs. dry sand produces a marked difference in chilling propensity. Mould moisture in contact with molten iron releases oxygen and hydrogen which are recognised as carbide stabilisers, and as such can have an effect on the structure of a thin section. Many investigators have shown that hydrogen, carbon monoxide and nitrogen are largely responsible for the expansion of cast iron. Plates and rings can exhibit swelling when cast in green sand. This can be prevented by pouring these castings in dry sand where mould hindrance would minimise if not eliminate the swelling. Swells are most troublesome in operations such as broaching and in other set-ups tooled for high production. Mould dilation in green sand results largely from the expansion associated with the solidification of cast iron and is not a simple ferrostatic phenomena. Control of moisture and the use of additives to improve the properties of the sand can improve this situation very much.

An example of the difference in tool wear that can occur on machining two batches of rings cast in core sand vs. two similar batches cast in green sand is depicted in Table III. Again, the results pertain strictly to the

TABLE II—TOOL LIFE TESTS ON GREY IRON RINGS

Batch	Pouring Temperature		Surface Machined*	Average Number of Ring Faces Machined	Volume of Metal Removed (cu. in.)†	Weight Remvd. (lb.)
	(°F.)	(°C.)				
A	2,650-2,670	1,455-1,410	As-Cast	10	15.7	4.0
			Pre-machined	35	54.9	14.3
B	2,570-2,550	1,410-1,400	As-cast	3	4.7	1.2
			Pre-machined	10	15.7	4.0
F	2,560-2,530	1,405-1,390	As-cast	5	8.6	2.2
			Pre-machined	20	31.4	8.1
H	2,630-2,600	1,445-1,425	As-cast	10	15.7	4.0
			Pre-machined	30	47.1	12.2
L	2,660-2,610	1,460-1,430	As-cast	16	25.1	6.5
			Pre-machined	38	44.0	11.4

COMPOSITION (%)

Batch	T.C.	Si	Mn	Cr	Cu	Ni	C.E.
A	3.45	2.06	0.70	0.16	0.12	0.03	4.13
B	3.39	2.09	0.75	0.23	0.10	0.04	4.09
F	3.39	2.15	0.81	0.18	0.08	0.04	4.10
H	3.42	2.04	0.69	0.17	0.08	0.03	4.10
L	3.40	2.19	0.81	0.16	0.09	0.03	4.13

\*—Facing cut of  $\frac{1}{8}$  in. depth made on 9 in. O.D.  $\times$  7 in. I.D.  $\times$   $\frac{1}{8}$  in. thick rings, as-cast surface. Then test repeated on the pre-machined rings removing metal from  $\frac{1}{8}$  to  $\frac{1}{4}$  in. below as-cast surface.

†—Based on cubic inches of metal removed for 0.020 in. wear land at 300 ft./min. cutting speed and a feed of 0.009 in./rev. Tool geometry of grade 44A carbide tools used is given in Table I.

TABLE III—TOOL LIFE TESTS ON GREY IRON RINGS

Type of Mould	Composition (%)					Average Number of Ring Faces Machined*	Volume of Metal Removed (cu. in.)†
	T.C.	Si	Mn	S	C.E.		
Green Sand	3.38	2.26	0.69	0.10	4.33	12	18.6
	3.56	2.26	0.68	0.11	4.31	19	29.5
Core Sand	3.41	2.26	0.74	0.11	4.16	27	43.5
	3.46	2.06	0.57	0.10	4.15	33	53.0

\*—As-cast surface machined to a depth of  $\frac{1}{8}$  in. on 9 in. O.D.  $\times$  7 in. I.D.  $\times$   $\frac{1}{8}$  in. thick rings individually cast in core or green sand.

†—Based on cubic inches of metal removed for 0.020 in. wear land at 300 ft./min. cutting speed and a feed of 0.009 in./rev. Tool geometry of grade 44A carbide tools used is given in Table I.

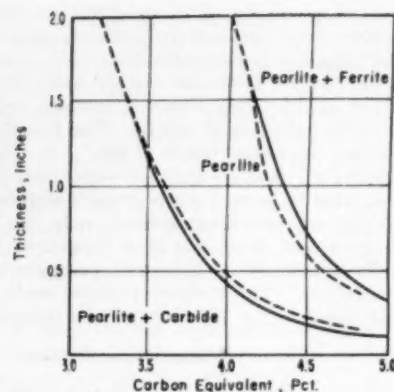
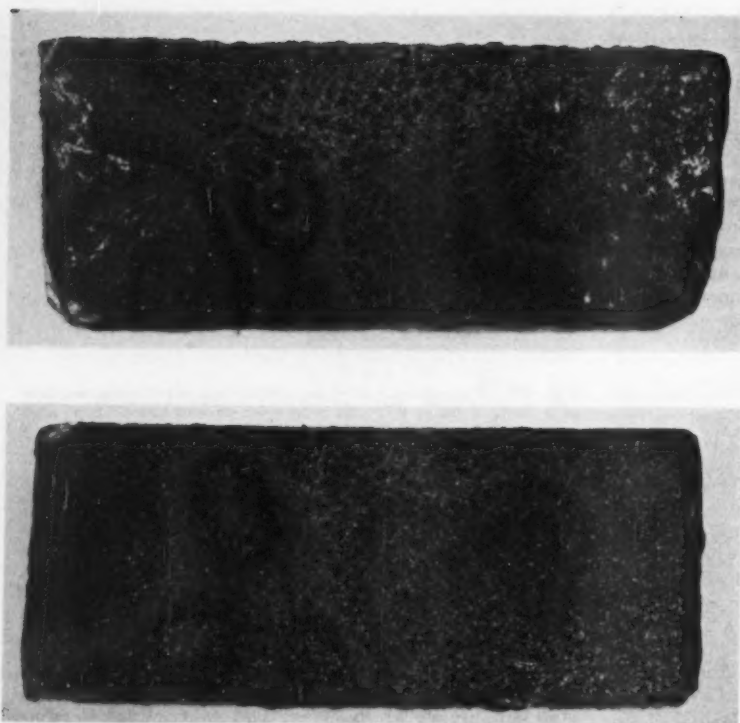


Fig. 4.—Section thickness, carbon equivalent and microstructure of cast iron, according to Schneidewind and McElwee.

Fig. 3.—Macrograph of  $\frac{1}{2}$  in. thick step bar cross section of 3.30% T.C., 1.87% Si iron cast in wet sand (top) and dry sand (bottom). Etched in picral.  $\times 3$

casting design under consideration, and comparison is made on a like chemical and thermal basis. For these class 25 irons, the machinability of the rings cast in core sand is 40–170% better than that of rings cast in green sand with controlled moisture. This difference was obtained in spite of the higher total carbon and carbon equivalent of the rings cast in green sand. Furthermore, the machinabilities of these rings are no better than the machinability of the rings cast in core sand by another producer, listed in Table II, which contain significant amounts of chromium, a known carbide stabiliser and detrimental to machinability. Since there seems to be little doubt that the moisture content of the moulding sand has an effect upon machinability, it is important to evaluate the role of mould moisture and to control it accordingly.

The effect of mould condition can be an important factor in malleable iron casting. Malleable iron rims are not always due to conditions of anneal; metal composition, melting practice and mould conditions are often responsible for certain types. In a metallographic study of malleable iron step bars, a marked reduction in graphite nodule count on the surface of some of the step bars was noted. This was definitely a mould effect, since these step bars were fractured prior to annealing, and the fractured edges were free of the rim upon examination after annealing. Only the mould surfaces exhibited the rim. These step bars were poured vertically in core sand moulds designed so that ten step bars could be poured in about 5 minutes while the metal temperature ranged between 2,650° F. (1,455° C.) and 2,609° F. (1,430° C.) (noble metal thermocouple). Constant-pressure lathe tests were made on  $\frac{1}{4}$  in. arbitration bars cast from the same ladles,

but the machinability ratings were determined after all or part of the rim was removed when the bars were first turned down to  $\frac{1}{4}$  in. diameter.<sup>12</sup>

### Inoculation

Inoculation practice has become popular as a means of overcoming the section sensitivity of cast iron. Referring to Fig. 4, it has been proven by experiment and extensive practice that the use of adequate additions of effective inoculants moves the left hand curve further to the left while affecting not at all the right hand curve. This is particularly important in the production of high strength irons with 100% pearlite structure. Not only is chill reduced but there is benefit from reduction or complete elimination of any eutectiform arrangement of the graphite without much coarsening of the refined graphite flakes. Somewhat differently expressed, the possibilities for the occurrence of chilled iron are reduced or confined to thinner sections. A section that exhibited eutectiform graphite is changed to one containing small random flakes and only one that after superheating was chilled will, after inoculation, display eutectiform graphite in a pearlite matrix.

Not only the presence of any hard carbides but also the uneven distribution of ferrite and pearlite at the surface of a casting are removed by effective inoculation (ferrite associated with eutectiform graphite). The form and disposition of these constituents have a very definite effect on the behaviour of the cutting tool during the machining operation, and the net result is a difference in the amount of metal removed for a particular degree of tool wear. Even where the graphite is relatively unaltered, but the pearlite is varied from fine to coarse, the machinability is affected in the same

way. In most cases the microstructure of the iron is determined by the mechanical property requirements of the casting, so that the structure for optimum machinability cannot be obtained. However, proper inoculation can be very important in correcting this situation.

The beneficial effect of calcium-silicon inoculation on machinability was studied in a large foundry producing an iron containing 3.35-3.50% carbon and 1.90-2.00% silicon. Each 4,000 lb. charge in the 60 in. cupolas consisted of 30% pig, 30% steel and 40% scrap, and the calcium-silicon was fed uniformly into the tapping stream. The machinability test rings of 7 in. I.D., 9 in. O.D. and  $\frac{1}{2}$  in. or  $\frac{1}{4}$  in. thickness were individually cast in core sand to eliminate the variable effect of moisture on the surface structure of green sand castings. The ring specimens were poured from 1,200 lb. ladles and at comparable temperatures. Before the machining tests were made, the rings were separated into appropriate batches of equivalent chemistry, the comparison with untreated iron being made within the carbon equivalent of 4.10-4.20%. The details of the tool life tests were described in a preceding section.

The results on machining the as-cast surface to  $\frac{1}{16}$  in. depth with grade 44A carbide tools at various speed are depicted in Fig. 5. Tool life was determined by measuring the wear on the flank of the cutting tool. The tests on machining successive rings being stopped when the wear land reached a width of 0.005 in. Tests stopped at this width of wear land correlate well with those run to 0.025-0.030 in. wear land, which represents complete failure for carbide tools. The merit of calcium-silicon inoculation in improving the skin structure of the ring castings, and hence machinability, is shown for all cutting speeds. The improvement in machining the  $\frac{1}{2}$  in. thick rings ranges roughly from 100 to 300% for the cutting speed range of 150-315 ft./min. The ability of calcium-silicon inoculation to reduce chill is shown by the fact that the  $\frac{1}{4}$  in. thick rings of the treated irons were as machinable as the  $\frac{1}{2}$  in. thick rings of the untreated irons. The  $\frac{1}{4}$  in. thick rings cast from untreated irons could not be machined because of excessive corner chills which immediately cratered the flank of the cutting tool.

The merit of inoculants in cast iron is proven if they alter the microstructure to the point that a definite improvement in machinability is realized. The type of graphite structure produced in a casting is primarily the result of the length of time the casting spends in going through the eutectic arrest. The longer this time, the coarser will be the graphite structure. Actually, as silicon is added to the iron-carbon system, the eutectic reaction occurs over a range of temperatures and also starts at a slightly higher temperature. The incomplete nature of the Fe-Si-C equilibrium system precludes definite conclusions, though recent work has established silicon carbide as a stable phase in the ternary equilibrium diagram. It appears that the addition of a silicon-bearing material to cast iron cuts through the silicon carbide region of the ternary, which on resolution precipitates graphite, thereby inoculating the iron. The mechanism of inoculation may very well be physical rather than chemical. If a chemical reaction is associated with an inoculation treatment, the reaction apparently is not one of deoxidation.

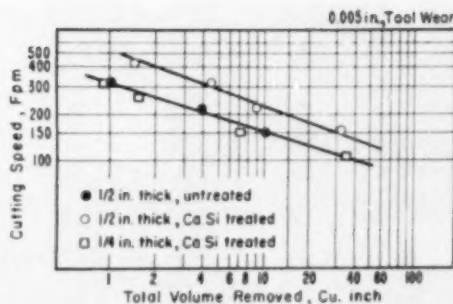


Fig. 5.—Tool wear data on machining  $\frac{1}{2}$  in. and  $\frac{1}{4}$  in. thick rings at depth of  $\frac{1}{16}$  in. below as-cast surface. Composition of iron: 3.35-3.50% T.C. and 1.90-2.00% Si.

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## News in Brief

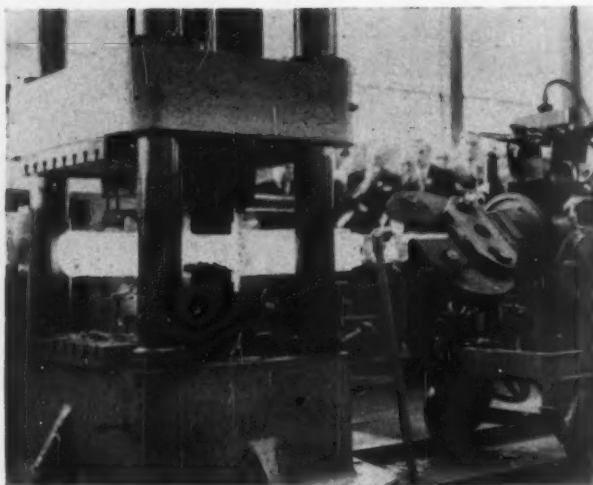
ALUMINIUM LTD., and The British Aluminium Co., Ltd., have announced the completion by their jointly owned company in Australia—Australian Aluminium Co., Ltd., of plans for a further expansion of its rolling mill and extrusion facilities in Sydney. These plans are being implemented nearly two years ahead of schedule. When the programme has been completed in 1963, the Australian Aluminium Co. will have the capacity to produce a total of approximately 20,000 long tons per annum of rolled products and 9,000 long tons per annum of extruded products.

A NEW company, D. A. Stuart Oil Co. (G.B.) Ltd., has been formed, as a wholly owned subsidiary of the Amber group of companies, to market in this country the products of D. A. Stuart Oil Co. Ltd., of Chicago and Toronto, widely known in North America as producers of metal working lubricants since 1865.

SCHLOEMANN A.G., Düsseldorf, in conjunction with Concast A.G., Zürich, are to build a vertical, continuous casting unit for the Aktiengesellschaft der Dillinger Hüttenwerke. The plant is to provide for the production of slabs up to 60 in.  $\times$  8 in. and is scheduled to go into operation early in 1961.

A PLATE mill complete with auxiliary equipment, built by Schloemann A.G., has recently been commissioned at Oxelösunds Järnverk AB., Sweden. The four-high reversing mill has a barrel length of 12 ft. and the designed output is 30,000-35,000 tons a month.





Close-up of the forging press and manipulator during a demonstration.

## Towards Automatic Forging

### Progress of B.I.S.R.A. Research Project

**M**ENTION of automation tends to conjure up visions of raw materials being fed into one end of a factory to emerge later at the other end as "millions" of identical products. At first sight the idea of introducing automation in forging seems rather ambitious, but such is the eventual aim of a research programme which has been carried out by the British Iron and Steel Research Association during the last few years, and which has resulted in appreciable progress towards the final goal. It should be stressed at the outset that the term forging here refers to the operation whereby a mass of red hot metal is shaped by successive blows from a hammer or squeezes from a press, using flat or curved tools in the main: closed die forging to produce relatively close tolerance products is not included. Production is on a jobbing or small batch basis and covers such items as billets, simple shaped shafts, collared shafts, rolls, rotors, etc., made to fairly crude dimensions, involving the machining away of large quantities of material at a later stage. The B.I.S.R.A. work has been mainly concerned with forging by means of a hydraulic press.

For the successful production of forgings of the type referred to above, a team of experienced operators is required at the press. It was, in the first place, a shortage of skilled labour and the increasing difficulty of recruitment for forging that prompted the B.I.S.R.A. forge mechanisation project. It was also apparent at that time—some five years ago—that there was a need for increasing mechanisation if the British forging industry were to be able to meet competition from the Continent with any hope of success.

In a report published last year\* on operational research studies carried out by B.I.S.R.A. in the forging industry, it is pointed out that a feature of the industry is the low utilisation of the actual forging equipment, which is partly caused by inefficient or insufficient furnacing, partly by inadequate handling equipment, and partly by other factors, including inadequate production programming. It is evident, therefore, that the full benefits of mechanising the forging operation

itself will not be achieved unless there is sufficient heating capacity available, and unless the scheduling of work in the furnaces is efficient.

The forging operation comprises a number of movements of the press and of the stock being forged. Stock movements may be vertical, horizontal or rotational, and a suitable combination of these with the movements of the press will produce whatever shape is required. The ultimate goal of the B.I.S.R.A. programmed forging project is to carry out the whole operation automatically by determining in advance the way in which the stock is to be worked and setting the sequence controls accordingly.

Following a study of existing forges which revealed the following shortcomings:

- (1) process and handling equipment too slow;
- (2) accuracy of control rather low; and
- (3) co-ordination between press and manipulator poor;

visits were paid to establishments abroad, particularly in the United States where mechanisation was furthest advanced. The outcome of these studies was a research programme divided into three stages: (a) improvements to the operation of the forging press; (b) the development of a suitable manipulator; and (c) the synchronisation of the action of the press with that of the manipulator.

The first target was to raise the efficiency of the forging press, using the B.I.S.R.A. 200 ton experimental press at the Hoyle Street Laboratories in Sheffield. By improvements in the hydraulic system, the stroking rate was increased to 60 cogging strokes per minute. Concurrently, a system of remote position control was developed which enabled the amount of squeeze to be predetermined to within  $\pm \frac{1}{16}$  in. The controls include provision for repetitive stroking of the press cross-head between fixed limits.

Attention was then turned to the manipulator. At the anvil rapid longitudinal, rotational and, possibly, lateral movements are required over short exact distances. The manipulator must, therefore, have high rates of acceleration and be able to grip the ingot at the ends,

\* J. Iron and Steel Inst., 1959, 190, 148.



and should lend itself to automatic position control. To achieve this, the gripping and moving parts of the manipulator must be as light as possible. Since it was considered essential that the manipulator should not be called upon to transport the stock from the furnace to the press in addition to holding and locating it during forging, the prototype was made rail-bound. Provision was made for longitudinal feed, for accurate radial movement through  $90^\circ$ ,  $45^\circ$ ,  $22\frac{1}{2}^\circ$  and  $7\frac{1}{2}^\circ$ , and for adjustable height. These movements had to be made rapidly and accurately and the design incorporated hydraulic actuation and electrical control of each motion. In order to make the moving parts of the manipulator as light as possible, all possible equipment was located on the floor with flexible connections to the manipulator carriage. Provision was made in the electrical control system for synchronising the movement of the press and manipulator, so that, as the press cross-head moved upwards on its return stroke, the stock was immediately moved to the next working position by the manipulator.

The next step was to "programme control" both press and manipulator. Investigations of metal shaping were therefore carried out to establish the precise sequence of squeezes and manipulator movements required for a particular forging operation. Previously these steps had been arrived at by empirical methods, and B.I.S.R.A.'s investigations constituted an attempt to rationalise procedure so that a predetermined programme could be formulated. As yet, theoretical schedules have been worked out for square bars only, but further work continues on other shapes.

An experimental control device has been produced for applying these programmes to the combined press and manipulator. Thirty identical panels are available on which every detail of the forging schedule are set up. In this schedule are given the upper and lower limits of the squeeze for every pass, the manipulator feed between strokes, and the manipulation necessary between passes. The control unit signals instructions to the press and manipulator in the appropriate sequence. This experimental set-up is flexible, but a simpler one with fewer panels is envisaged for actual forging operations.

At a recent demonstration an 8 in. sq. ingot was cogged down in twenty-three passes to a forging having three sections, each 20 in. long, with cross-sections  $6 \times 6$  in.,  $4 \times 4$  in., and  $3 \times 3$  in. This programme was completed to a better-than-average standard of accuracy in  $4\frac{1}{2}$  minutes—about twice as fast as the best skilled forging crew would be capable of doing—and even faster working will shortly be practicable. Particularly important is the accuracy possible as regards the concentricity of the three sections relative to each other.

The present position of the B.I.S.R.A. programmed forging project may be summarised as follows:

- (1) Precision control of pressing has been achieved and the equipment is already in use in industry.
- (2) Fast mechanised handling with precision control: this has been completed and is in commercial development. One set of equipment is ready to go into operation.
- (3) Joint control of press and manipulator: this has also been completed and is in commercial develop-



General view of the forging press and manipulator and the associated hydraulic and electrical gear.

ment. Delivery of the first set is expected early next year.

- (4) Forging by pre-set programme: this has been completed for simple shapes, but work continues on more complicated ones.
- (5) Self programming: this is of course the ultimate objective, and work is continuing with a view to developing a suitable system.
- (6) Equipment going into service has been designed so that more advanced stages can be added at a later date.

When the present programme was started some five years ago the United States held the lead in forge mechanisation, and as far as the industry as a whole is concerned this is probably still true, but Western Europe is catching up. With the development of programmed forging, however, B.I.S.R.A. may now be considered to lead the world as far as remote control (beyond the laboratory stage) is concerned. This advance in forging techniques comes at a time when many firms are already planning to re-equip their forges, in some cases replacing machines dating back to World War I. At the moment there are probably more forging presses on order in this country than at any previous time: this development of automatic working therefore offers the forging industry a great opportunity to put itself ahead of foreign competition.

### Limestone for Sintering

ADAM LYTTHGOE, LTD., of Culcheth, Warrington, producers of lime and fertilisers, have secured a contract for the supply of 180,000 tons of limestone grit to John Summers & Sons, Ltd., at Hawarden Steel Works, Shotton, Cheshire. The material, conforming to precise grinding specifications, will be used in conjunction with Summers' new sinter process.

# Stainless Steel Supersonic Aircraft

## Builder and Steelmaker Co-operate

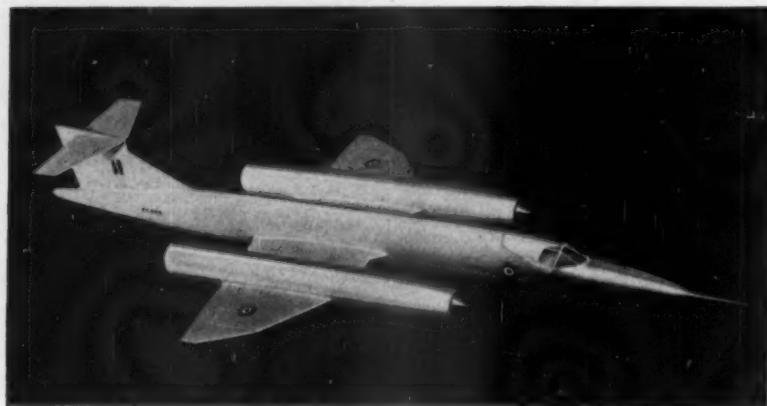


Fig. 1.—Model of the Bristol Type 188.

**B**EHIND almost every engineering achievement there lies a story of co-operation; co-operation between the designer and his manufacturing team, on the one hand, and the metallurgist—in the wider field, the materials engineer—and the supplier of materials, on the other. An interesting illustration of this point concerns the use of stainless and alloy steels in the construction of the Bristol Type 188 supersonic research aircraft due to fly soon at speeds exceeding 1,500 m.p.h. A general idea of the configuration of the Type 188 is given by Fig. 1, and the aircraft itself is seen in course of construction in Fig. 2.

One of the effects of flight at such high speeds as are envisaged for the Type 188 is the subjection of the aircraft structure to temperatures much higher than prevail in conventional subsonic or low transonic machines. For example, under the combined effects of kinetic heating and the heat generated by the engines and other installations, temperatures of about 250° C. are likely to endure for such periods that the thinner portions of the structure will reach a steady condition. In these circumstances, aluminium alloys would be unsuitable, and the use of carbon and alloy steels would pose formidable questions of manufacture, condition of supply, and protection against corrosion for the service life of the aircraft. At the relevant time, indigenous production supplies of the sheet form of titanium were restricted to the commercially pure varieties; British alloy sheet materials were still in the development stages, and foreign supplies barely out of them from the aspects of demonstrated consistency, adequacy of sheet size, thickness, tolerance, flatness, etc. Accordingly, it was decided that the Type 188 should be of welded stainless steel construction, and from that point onwards the progress of the development of the aircraft was marked by the closest of co-operation between Bristol Aircraft Ltd., and Firth-Vickers Stainless Steels, Ltd., firstly, in reaching decisions about the materials to be employed for the major structural parts, and, secondly in arranging

the supply of the selected materials in the correct and precise condition as regards chemical composition, strength and size.

### Sheet and Plate

The importance of material in sheet form as constituting the envelope surface of any aircraft needs no emphasis, for Fig. 2 demonstrates it clearly. Generally speaking, the stainless steel industry's main aircraft specialisation has probably been the supply of bar, forgings and castings for use in gas turbine engines, together with a certain amount of heat resisting steel sheet for their combustion chambers. The industry's sheet plant has, therefore, been planned almost wholly against commercial applications—mostly in the softer forms—and the needs of a project like the Type 188 for relatively small quantities of specialised forms of stainless steel sheet represented an appreciable disturbance of the industry's normal commercial activity.

The choice of the particular variety of stainless steel to be used had to be made on the basis of the following criteria:—

- (1) highest possible elastic modulus to safeguard structural performance in overall stiffness and in local stiffness in the design of compression structures;
- (2) highest possible strength consistent with reasonable ductility and freedom from stress-corrosion troubles;
- (3) reasonable uniformity of mechanical properties as between longitudinal and transverse directions of loading, and also as between loading in tension and compression;
- (4) simplicity of any heat treatment procedures necessary to restore adverse effects of cold working consequent upon the manipulation in aircraft fabrication processes;
- (5) availability of reasonably large sizes of sheets, to avoid unduly high numbers of joints, with a good

standard of flatness, of surface finish, and of conformity to closer tolerances than are commercially acceptable; and

- (6) minimum possible drop in properties at the operational temperatures involved.

The procedural difficulty that no suitable national material specifications existed and that special inspectional controls would be necessary was accepted as appropriate to such a research aircraft.

The precipitation-hardening stainless steels were still in their infancy in this country and consideration of them was necessarily limited. Of the remaining materials, the complex 12% chromium stainless steel of the FV.448 type—originally developed as a creep-resisting steel for gas turbines—will meet most of the above requirements handsomely, but considerable difficulties arise in the manufacture of flat sheets of a sufficient size and in a very high strength heat treated condition. Very little forming can be done in this hardened condition and stretch flattening is impossible. The surface finish of the sheets as received is that appropriate to a hot rolled and hardened and tempered steel product necessarily manufactured as individual sheets, and the flatness only just adequate after hammer flattening by the maker. Nevertheless, this steel is most attractive structurally, and hence it has been used wherever these limitations are acceptable. For some parts it is regarded as machining plate stock, to be subsequently milled and ground as appropriate to achieve the necessary close tolerances. Where thinner forms of this FV.448 material are applicable, they are manufactured by the normal hot and cold rolling processes, with intermediate stretching at the appropriate stages to achieve the best flatness, prior to heat treatment in vertical ovens. Even these thinner materials are sometimes skin-ground all over before use.

For the major outside envelope of the fuselage structure where double curvature forming is involved it was decided that FV.448 steel would be unsuitable, and eventually the conclusion was reached that it was essential to utilise the inherent advantages of a cold rolled form of material for this duty, and a titanium-stabilised 18% chromium, 9% nickel steel (Firth-Vickers F.D.P.) was chosen for such duties, accepting its somewhat less satisfactory physical properties.

However, merely to say that plate and sheet materials for particular areas of application could then be selected on such general considerations would convey a very inadequate idea of the technical effort which necessarily still required to be devoted by both Bristol Aircraft and Firth-Vickers to the detailed assessment of the strengths of the material to be used in the structural design calculations. In the initial stages the airframes designer drew very heavily on the accumulated experience and data available from the laboratory facilities of Firth-Vickers, in respect of the general characteristics and potentialities of the various materials under scrutiny. The second stage became one of assessing how the

detailed properties would be influenced by the particular manufacturing methods to be adopted, and most of this work was carried out at Bristol.

The hard F.D.P. cold rolled material furnishes a good example of the care necessary to achieve the desired results. This steel exhibits some directionality in elastic properties, particularly in regard to proof stresses in compression and tension. Although for many applications this difference is of no concern, airframe design requirements are such that it had to be extensively investigated, and an improvement was obtained by heat treatment at 450–550° C.

Again, some very detailed knowledge of thickness tolerances being achieved on sheet and strip was desirable in order to safeguard weight. This was only to be achieved by Firth-Vickers taking measurements during representative production runs for Bristol to assess statistically, before the full quantities of material could be ordered with assurance. Moreover, uniformity of mechanical properties depends on close control of thickness tolerances since these govern the amount of cold rolling applied. It was necessary to investigate the metallurgical and strength characteristics appropriate to the hardened and tempered FV.448 sheet and plate for use as machining stock; earlier production of this steel having been confined to bars and forgings. Apart from the establishment of typical design strength and modulus values under conditions of both tensile and compressive loading in both directions of the sheet, decisions were taken regarding the performance of the steel under combined stress and corrosion, the best heat treatment to ensure a safe metallurgical condition, and the precise composition to be adopted in order to keep the hardness of the welds down to a reasonable level. In fact, a carbon content somewhat lower than that customary in the bar and forging form became a special requirement for the sheet and plate form, and a different heat treatment had to be used. With the FV.448 material it was also proposed to apply a stress-relieving treatment as a last operation before component assembly, to counteract the effects of cold-work during manufacture of the aircraft parts and to

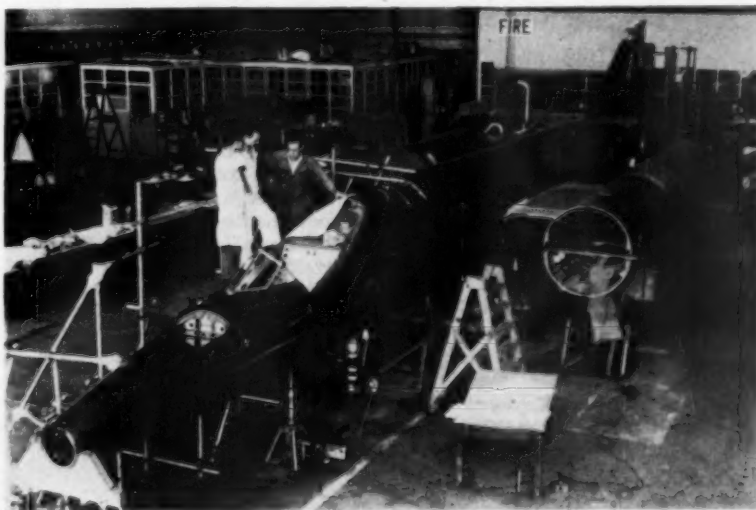


Fig. 2.—The Type 188 under construction at Filton.



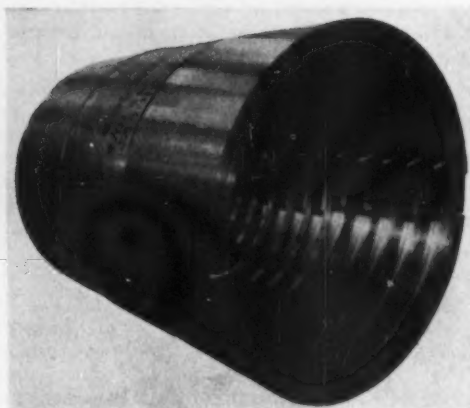


Fig. 3.—Three forgings assembled to form a nacelle.

realise by selection the best possible elastic modulus. So here again there was the situation that material was to be supplied in one condition to be utilised in a slightly different one, and the corresponding necessity for the two firms together to evolve the specification governing the acceptance of the material, both initially at Firth-Vickers' works and finally by selection at Bristol for incorporation into the aircraft.

Achievement of the necessary standards of flatness for the machining operations was no small matter either. It was soon established that the normal methods of manufacture did not yield an acceptable standard and that, as FV.448 is a stiff material, stretch-flattening is not a practicable proposition. Roller-levelling yields some improvement in converting hot rolled and heat treated sheets into a shape with less random buckling and with more nearly uniform curvatures, but the degree of flatness is still not sufficient. After much experimentation in the works of Firth-Vickers, one of their parent companies (English Steel Corporation), and a specialist equipment manufacturer (Messrs. Joshua Bigwood), this problem was solved by adopting some of the techniques of armour-plate manufacture, mainly a combination of hardening and tempering by dipper-grid and block methods, with subsequent hammer-flattening. An interesting sidelight is that no specification for aircraft sheet material has ever contained any quantitative requirement for flatness as a characteristic, but here it was necessary to evolve one—in terms of both general and local flatness—in order to safeguard the interests of both firms. Another, but later, interesting innovation was the introduction in both companies of ultrasonic methods of inspection of the unusual form of plate material, in order to ensure regularity of metallurgical cleanliness.

#### Bar

In the form of rolled bars, FV.448 is pre-eminently suitable for the general needs of Type 188 aircraft. Fortunately, in this instance, the peculiar difficulties associated with the sheet form do not apply. In bar form, it is the 18/8 material that is of limited application—due to the strength being that of the softened condition—to those parts where compatibility with the corresponding sheet form, low magnetic properties, higher corrosion resistance or more ready weldability is desirable. Thus FV.448 is chosen for the majority of parts produced from

bar stock (including fasteners) and in a wide range of sizes from "billets" for subsequent working at about 3 in. diameter to wires at about 0.1 in. diameter.

Apart from the use of FV.448 bar material in the general strength range of 70–80 tons/sq. in., the Type 188 aircraft was considered to justify the use of ultra-high-tensile low alloy steels, in the strength ranges 90–100 and 110–120 tons/sq. in., for bolts, bolt-like parts and special forgings. The particular virtue of ultra-high-tensile steels may be expressed as high strength for weight for size; for example, the use of them for bolts in lugs and fittings reduces the size of the fittings also and saves weight as well as room.

While the FV.448 bar material was supplied directly by Firth-Vickers, they arranged the supply of the forging form of it and of the U.H.T.S. in both bar and forging form from their parent companies, English Steel Corporation, Ltd., and Thos. Firth and John Brown, Ltd.

#### Forgings

Some of the forgings are somewhat unusual and merit a brief description. As can be seen from Fig. 2 the inner wing is somewhat thin, and an unusual method is used for the transfer of the loads from the outer wing across the engine nacelle. The mid-section of each engine nacelle consists of three machined cylindrical forgings of FV.488 steel bolted together end-wise, as in Fig. 3. These forgings were developed in co-operation with Firth-Vickers and Firth-Brown, and are believed to be the largest aircraft steel forgings made to date. The weight of each as-forged is between 3 and 4 tons, and as-supplied, "gashed" as shown in Fig. 4, it is about 21 cwt. Inspection at Firth-Brown is most comprehensive, including ultrasonic and electromagnetic inspection and chemical etching for the detection of any irregularities. Finished weight after final machining varies between 171 and 270 lb. for the three forgings.

Type 188 also utilises what are believed to be among the first ultra high-tensile steel forgings to be embodied in a British aircraft. These are in 110–120 tons/sq. in. material in a composition generally similar to En 40 C and are made by Firth-Derihon. They form the attachments for the outer wing and are pocketed into the nacelle: a group of them is shown in Fig. 5.

Another somewhat unusual forging in FV.448 is used for the keel boom of the aircraft and here the problem is one of length required in relation to heat treatment equipment available and the achievement of a straight forging after the heat treatment processes. In its final form for incorporation into the aircraft, the length required is a little short of 27 ft. and the thicknesses of the angle section vary from 0.45 in. to 0.1 in. and from 0.3 in. to 0.1 in. on the base and vertical flanges, the corresponding flange widths being of the order of 3 in. and 4 in., respectively. Making allowances for clamping and location during machining (as shown in Fig. 6), for extraction of test pieces, and for the extra length required for manipulation during manufacture at English Steel Corporation, the original length is some 39 ft. and the original section is  $4 \times 3\frac{1}{2}$  in., locally reduced to match the capacity of the resistance-heating equipment available. Rolled bar is used as the stock, and the hardening operation is conducted in a tunnel furnace specially constructed by E.S.C., the main heating being by resistance heaters arranged round the walls of the tunnel. Straightness is maintained by a load hydraulically applied to the bar in its expanded





Fig. 4.—Individual forging supplied in the gashed condition.

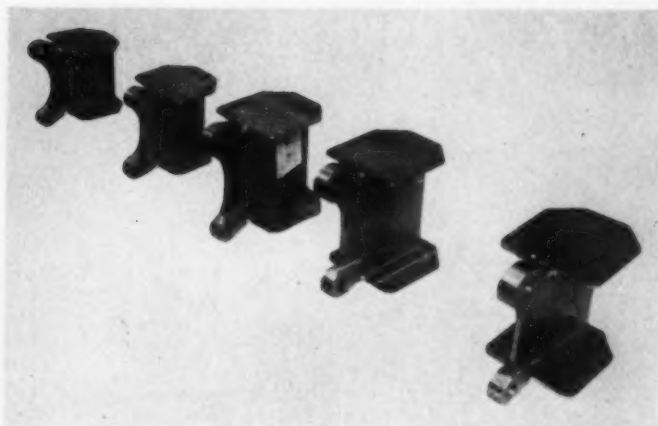


Fig. 5.—Group of ultra-high-tensile wing attachment forgings.

condition and maintained until it has cooled below its critical temperature. For tempering, the subsidiary resistance heaters are not used, the sole source being resistance-heating of the bar itself; the same hydraulic loading is used to maintain straightness.

#### Fasteners

Early in the development of Type 188, serious consideration was given to the choice of materials for the various forms of fasteners required. In particular, it was desired to attain for the strength of solid rivets a considerable advance on the 35–45 tons/sq. in., typical of materials conventionally used for the joining of ferrous materials. After trials of several possible materials it was found that FV.448 performed very well in the fully hardened and tempered condition at 70–80 tons/sq. in. and that the rivets could be set relatively easily by squeeze or percussion methods, in sizes up to  $\frac{3}{16}$  in. diameter. The limitation was in the load that could be applied and the ease and accessibility with which it could be brought to bear. Fig. 7, shows a selection of the fasteners used for Type 188, including the solid rivets machined by Messrs. Brown Bros.

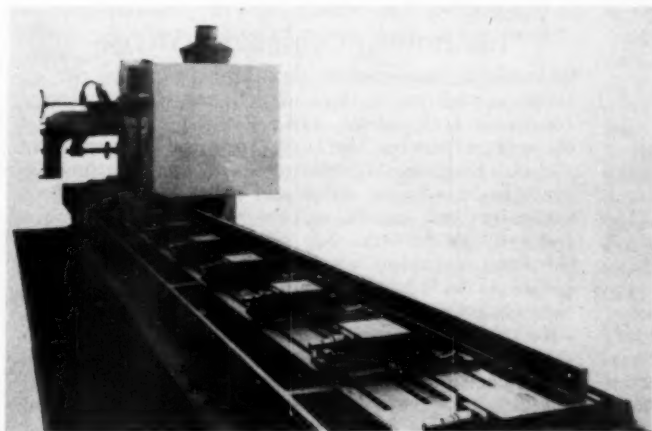
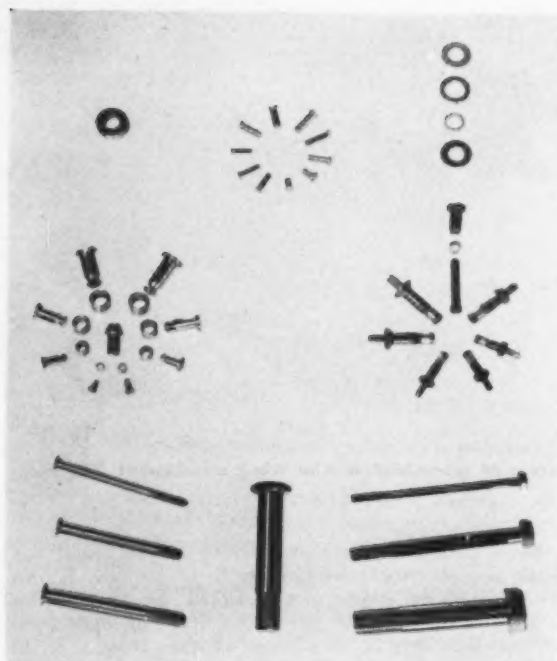


Fig. 6.—Keel boom forging supplied in the rough machined condition.

For applications where accessibility did not permit the loads required for solid-riveting to be applied, Brown Bros. Hi-Shear rivets were selected for their easier setting characteristics. These also were machined from FV.448 bars or rods in a range of sizes from  $\frac{1}{4}$  in. to  $\frac{5}{16}$  in. diameter; for their collars a special titanium-bearing 13% chromium steel (Firth-Vickers FI.(G)) was adopted. This steel had been specially developed for golf club heads and has excellent formability.

Sufficient has already been said about fasteners to emphasise that when an aircraft like Type 188 demands unusual materials for the major structure, it is equally essential to develop a full range of fasteners in compatible materials. Then the criteria of performance for the needs of the specialist fastener manufacturer are often additional to those for the manufacture of the aircraft itself. This was illustrated in the supply of coils of wire for the three-piece "blind" fastener selected, Messrs. Aviation Developments' Jo-bolt. In order for the manufacture of the bolt and nut portions of it to be undertaken in the FV.448 material using the same high-speed cold-heading machines as are used for orthodox alloy steel materials, it was essential to control very closely the strength and condition of surface finish and of its lubrication. To achieve this control, close liaison was necessary with Firth-Vickers who supplied the cogged billet and with Messrs. Hemmings—the specialist wire drawers—as well as with Aviation Developments themselves.

Rather similar considerations prompted the supply of 3-in. billet material, from Firth-Vickers of FV.448 and from Firth-Brown of the ultra-high-tensile steel, to Unbrako Socket Screw Company for the manufacture of the many special bolts and screws required. In view of the importance they attach to surface finish and its final lubrication, and of the close tolerances they require for bolt and screw manufacture, their practice is to undertake all final rolling and drawing operations in their own mills.



Top left and right : pre-load indicating washer in FV.448 type material ; component parts on right, assembled washer on left.

Top centre : solid rivets in FV.448 type material

Middle left : Hi-Shear rivet components ; pins in FV.448 type material.

Middle right : Jo-bolt components ; " nuts " and " bolts " in FV.448 type material.

Bottom : typical structural bolts, showing countersunk, sheer and tension types in FV.448 and U.H.T.S. type materials.

Fig. 7.—Range of fasteners used on Type 188.

### Tubes

Type 188 utilises austenitic stainless steel piping for the conveyance of hydraulic power to its undercarriages, flaps and power-operated controls. Mainly it is in the soft condition but, where geometry permits, a harder drawn variety is used. The main needs are resistance to high working pressures, good fatigue performance under pulsating pressure, minimum weight, and ease of manipulation. To provide the best performance a very high standard of finish—both in the bore and externally—and close adherence to minimum tolerances are the primary requirements. The achievement of these desirable standards has been a matter for detailed discussion with the specialist tube makers, Messrs. Aceles and Pollock, since it is largely the final tube-drawing that conditions these factors. The tubes are drawn from hollows extruded by Chesterfield Tube Company, from, it is believed, Firth-Vickers F.D.P. austenitic stainless steel.

### Materials Development

Extraordinary difficulties are faced by the aircraft manufacturer in adapting new materials to the specialised design requirements of a Mach 3 aircraft. Having chosen

a material on the basis of such evidence as is available, often incomplete at the time, production and fabricating experience must be obtained by a series of carefully planned development trials. In this instance there was little previous experience of producing and handling high strength steels in the form of flat sheets. The choice of FV.448 on the basis of its physical properties gave rise, later on, to some manufacturing difficulties in regard to the flat rolled products, but proved to be an admirable decision in other ways.

The technical advisory staff of Firth-Vickers Stainless Steels, Ltd., was greatly involved in this work. The contribution they made might have been described at times as a form of negative salesmanship, since every conceivable limitation of the chosen stainless steels was brought forward and thoroughly investigated, at Bristol or at Sheffield. As with all such projects, inevitably a stage is reached when the teams find themselves committed to a particular set of manufacturing proposals. Notwithstanding that improved materials might be coming along in the steelworks, under the stimulus of the limitations revealed by the early trials, these must be put aside. In this sense the high strength stainless steels FV.520 and FSM.1 were developed by Firth-Vickers as a direct consequence of the decision to go ahead with the Type 188, and the experience gained in these development trials, but when they were perfected it was too late for them to be incorporated in this aircraft.

### Union Carbide Aids Research

To encourage and advance research in the field of chemistry, the Chemicals Division of Union Carbide, Ltd., which has a major petrochemicals plant located at Hythe, Hampshire, is making a grant of £2,150 per annum for three years to Southampton University and will later consider an extension of this grant. The grant will provide for a research studentship and a research fellowship in synthetic organic chemicals in the university's department of chemistry. The studentship will assist promising students chosen by the university to work for a master's or a doctor's degree and the fellowship should stimulate a research atmosphere. The Alloys Division of the company is giving similar support to other universities, notably, fellowships of £450 per annum to Sheffield University and £450 per annum to the Royal College of Science, Glasgow.

### Instrument Companies Merger

CAMBRIDGE INSTRUMENT CO., LTD., have announced 100% acceptance of their offer to the shareholders of Electronic Instruments, Ltd., Richmond, Surrey, and the merger between the two companies has been completed. Electronic Instruments, Ltd., will continue to trade under its own name and trade mark, but both companies will benefit by the co-ordination of their research, production and sales facilities. Mr. Paul Goudime, managing director of Electronic Instruments, has joined the Cambridge board. Dr. P. Dunsheath, C.B.E., chairman, Cambridge Instrument Co., Ltd., Mr. H. C. Pritchard, managing director, and Mr. W. E. Lamb, director, have joined the board of Electronic Instruments, Ltd., of which Mr. A. C. W. Norman, O.B.E., will continue to be chairman, Mr. Paul Goudime, managing director, and Mr. D. A. Pitman, sales director.

# Twin Chamber Electric Batch Furnaces for Heat Treating Aluminium Wire



General view of the installation. A load of coils is being withdrawn from the end furnace, and loads awaiting treatment may be seen on the loading tables between the furnaces.

**A**LTHOUGH continuous or semi-continuous furnaces are used for many applications, the batch method has generally been found to be quite satisfactory for the heat treatment of aluminium at various stages in its production. Three electric batch furnaces, of the twin chamber type, built and designed by G.W.B. Furnaces, Ltd., of Dudley, Worcs., are installed in the works of Aluminium Wire & Cable Co., Ltd., at Port Tennant, Swansea. This company was formed in 1946 to specialise in the production of aluminium wire and stranded conductor for overhead power transmission and distribution lines. The company was formed as a joint project resulting from the co-operation of several large companies who pooled their resources to provide maximum facilities for research and development. Included in the present range of products are hot rolled rod; solid and stranded conductors; round wire in coils, on reels, or in straight lengths; wire in rectangular and other sections; rivet wire; nail wire; flattened wire, etc. Materials processed comprise various grades of aluminium, from commercial purity to super purity, and a wide range of aluminium alloys, delivered to the works in the form of wirebars, 4 in. square and 9 ft. long.

A high-speed semi-continuous rod mill progressively reduces the wirebars, preheated in electric resistance furnaces, until the required size of round rod—usually  $\frac{3}{8}$  in. diameter—is obtained. The potential output of this mill is 30,000 tons a year. Wire drawing machines—including heavy duty single die machines, multi-die machines for drawing intermediate sizes, and multi-die fine wire machines—draw the wire to the finished sizes, the normal range being from  $\frac{3}{8}$  in. diameter to 0.0052 in. diameter. Intermediate and final annealing and heat treatment, essential processes in the production of many grades of aluminium and aluminium alloy wire, are carried out in the G.W.B. furnaces. The various purities of aluminium and certain aluminium alloys derive their strength from work hardening during drawing, and may be softened by closely controlled

annealing at temperatures of the order of 380° C. Other alloys derive their strength from heat treatment after drawing. Such alloys fall into two categories, the first of which harden at room temperature after solution treatment and those which require a further heat treatment, known as precipitation treatment, to attain maximum strength. The temperature of the solution heat treatment varies according to the composition of the alloy being processed, but is generally in the region of 500° C. A feature of this process is the rapid quench that is required immediately the material is withdrawn from the furnace, so as to retain in solution those alloying elements which have been taken into solution by the heat treatment. Care is taken to ensure an even, speedy quenching to avoid the distortion and uneven physical characteristics which would result in the metal in the event of uneven quenching. Precipitation hardening is normally carried out within the range 170°–190° C. For this low temperature heat treatment the power input to the furnace is reduced from the maximum available by means of delta/star switching, which has the effect of reducing the power to one-third. This power reduction when operating at the lower temperature range, during which less power is required, assists in reducing wear on the furnace contactors.

The three G.W.B. furnaces are installed in a heat treatment line and are served by a single Gibbons-van Marle charging machine which traverses along a track running the length of the treatment line. This machine is all electric and has two charging arms with a run-out of 14 ft. 3 in. and capable of handling a load of 1½ tons. The maximum discharge speed is 200 ft./min., with an average speed of 100 ft./min., and the lifting motion takes 10 seconds. This arrangement allows a speedy quench when solution treatment is being carried out. The traversing movement of the charging machine is approximately 100 ft./min. Between the furnaces and in line with them are a series of charge tables, the tops of which are grooved to accommodate the arms of the charging machine. The overhead crane is only used



for placing the charge skips on the loading tables initially and removing the heat treated charge from them for transfer to another part of the works, and for lowering the charge into and removing it from the quench tank when solution heat treatment is being carried out. There are two quench tanks sunk into the floor, on the inside of the charging machine track, in front of the first and third furnaces.

The first two furnaces were installed at the end of 1949, and to cope with increased production a third was subsequently installed. The heating chambers are 12 ft. long  $\times$  4 ft. 6 in. wide  $\times$  3 ft. usable height, and the walls are lined with heavy aluminised mild steel plate, while specially moulded refractory bricks form the roof arches. These linings are backed by insulating bricks and semi-rigid slab type insulation to reduce heat losses to a minimum. Bonded refractory and semi-refractory brickwork lines the heat resisting steel hearth tracks which accommodate the arms of the charging machine. Three rows of heat resisting charge supports extend the full length of each chamber and are provided with holes designed to ensure efficient air circulation under and vertically through the aluminium coils.

The heating chambers are rated at 144 kW. each, in two independent automatically controlled zones. This rating is reducible by delta/star switching, working in conjunction with temperature controllers, for use during soaking periods or for low temperature treatments. Each zone has its own forced air circulation system provided by fans directing an air current through heating elements which are suspended from the furnace roof, and are of nickel-chromium strip in sinuous form arranged edge-on to the air flow. Aluminised mild steel baffle plates separate the heating elements from the chamber, thus preventing direct radiation onto the sides and top of the charge; these baffles are designed to direct the high speed air flow through the elements, under and up through the charge. Each of the multi-blade high velocity fan units fitted in the roof is mounted on a high tensile shaft carried upon heavy ball and roller bearings. A heat dissipating rotor fitted directly above the furnace top plate ensures permissible bearing temperatures, and the shaft is driven through vee ropes by an independent, totally enclosed motor mounted as a unit on the top beams of the furnace casing.

Each chamber has an independent steel-encased, fully-insulated and balanced door; the doors are driven automatically by an electric motor, and operation is by push button control from the furnace front. To cut out heat losses the doors are arranged to clamp automatically against the frontplate when closed.

The number of alloys requiring different treatments, and the variety of heat treatments carried out, necessitate efficient temperature control for each furnace. This is provided automatically by a G.W.B. totally enclosed instrument cubicle housing four potentiometric non-indicating controllers, two potentiometric 2-point recorders—which enable a visual check to be made at any time during the process cycle and automatic time switches. The necessary switchgear is contained in another G.W.B. cubicle housing the four furnace contactors, delta/star fan motor starting contactors, etc., and the rating reduction switches. Safety contacts are fitted to the recorders so that the main electric supply is cut off automatically in the event of accidental overheating in any zone.

## Personal News

IN order to provide for the evolution of the board of Woodhall-Duckham Construction Co., Ltd., the following changes and additions have been made with effect from August 1st, 1960: Mr. J. SIMPSON to be designated deputy chairman (group) with overall responsibilities regarding the growing number of subsidiary companies; Mr. E. N. WENBORN to be appointed vice-chairman with particular responsibilities relating to the company as a contracting unit; and Mr. C. D. MUNTZ to be appointed a joint managing director. The following three members of the staff have accepted invitations to join the board: Mr. A. F. COTTRELL to be appointed director-in-charge, operating department; Mr. H. E. DYBLE to be appointed director-in-charge, construction department; and Mr. R. O. RICHARDS to be appointed director-in-charge, design and development department.

G.W.B. FURNACES, LTD., announce that following the untimely death of Mr. A. V. FRANCIS, their furnace division manager, the following appointments have been made. The new manager of the furnace division will be Mr. W. L. HARRISON, who has spent many years in the furnace industry, particularly on the melting side. In future, the division will operate as two units, one dealing with melting and one with heat treatment processes. Mr. J. SIMPSON will be sales manager (melting) and Mr. J. NICHOLLS sales manager (heat treatment). Both Mr. Simpson and Mr. Nicholls have been with the company for many years, the former for some time being manager of the Canadian branch. Mr. A. HEAD, for many years chief draughtsman, has been appointed chief designer.

DR. G. L. J. BAILEY has relinquished his position as superintendent of the Mond Nickel Company's Development and Research Laboratory in Birmingham in order to transfer to London to become manager of research. Mr. E. J. BRADBURY has succeeded Dr. Bailey in Birmingham.

TUBE INVESTMENTS, LTD., announces that Mr. W. W. HACKETT, Snr. C.B.E., on retiring from the chairmanship of Accles & Pollock, Ltd., after 61 years' service with that company, which he helped to found, has accepted an invitation to become its first president. Mr. Hackett was also a founder member of Tube Investments, Ltd., and served for many years as a director.

MR. W. K. WHITEFORD, of Pittsburgh, president of Gulf Oil Corporation, has been elected a director of The International Nickel Co. of Canada, Ltd.

MR. W. H. EVERARD, deputy general manager of the foundry division of Edgar Allen & Co., Ltd., has been elected president of the British Electric Steel Makers' Guild. Mr. Everard, one of the pioneers of oxygen-lancing in the electric arc furnace was elected secretary of the guild in 1958, and vice-president in 1959.

SIR WILLIS JACKSON, F.R.S., director of research and education of Associated Electrical Industries (Manchester) Ltd., is to return to academic life as professor of electrical engineering at Imperial College, University of London.

MR. T. H. COOK has been appointed chief applications engineer of The Morgan Crucible Co., Ltd. Mr. Cook was previously carbon department technical sales promotion manager, and has travelled widely for Morgans.



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# NEWS AND ANNOUNCEMENTS

## The Iron and Steel Institute

### NEW GOLD MEDAL AWARD

THE council of The Iron and Steel Institute has established a new gold medal award, to be known as "The Medal of The Iron and Steel Institute." This medal is to be presented on special occasions to societies which have, in the opinion of the council of the Institute, rendered exceptional service to science or industry over a lengthy period. The first award of the medal has been made to the Royal Society, in recognition of its unique service to science over a period of three hundred years. The medal was presented by the Hon. R. G. Lyttelton, past-president of The Iron and Steel Institute, at the recent centenary celebrations of the Royal Society.

### PRESIDENT-ELECT

At a recent council meeting it was unanimously agreed to propose Sir Charles Goodeve, O.B.E., F.R.S., for nomination as president-elect of the Institute at the autumn general meeting. Sir Charles will succeed Mr. W. F. Cartwright as president at the annual general meeting, next May, and will thus be in office at the time of the proposed special meeting in the United States in October, 1961.

Born and educated in Canada, Sir Charles was in effective control of the research and development programme of the Royal Navy during the later years of the war and was knighted for his services in January 1946. Sir Charles left the Admiralty to become director of the British Iron and Steel Research Association on its foundation in 1945. In this position he became an honorary member of council of the Institute in 1945 and was elected a vice-president in 1956.

## The Beilby Medal and Prize

SINCE 1930, at intervals of one or more years, awards have been made by the Administrators of the Sir George Beilby Memorial Fund, representing the Royal Institute of Chemistry, the Society of Chemical Industry and the Institute of Metals. Sir George Beilby had been president of each of these three bodies, and they jointly sponsored the appeal for subscriptions whereby the fund was raised as a memorial to him after his death in 1925.

The awards are made to British investigators in science in recognition of independent and original work of exceptional merit, carried out continuously over a period of years and involving the development and application of scientific principles in any field related to the special interests of Sir George Beilby, i.e. in chemical engineering, fuel technology or metallurgy, in their modern interpretations. The awards are intended as an encouragement to younger men and women (preferably under age 40) who have done distinguished work of practical significance in any of these fields.

In 1959 two awards, each of 150 guineas, were made. It was then agreed that no further award would be made before 1960. The Administrators have decided that henceforth each award shall consist of a gold medal as well as a substantial sum of money, and shall be known as "The Beilby Medal and Prize" and specified as being "For Advancement in Science and Practice." Such an

award will be offered at intervals of two years, but more than one may be made on the same occasion if there are several candidates of sufficiently outstanding merit.

Consideration will be given in due course to the making of an award (or awards) from the Fund in 1961. Outstanding work of the nature indicated may be brought to the notice of the Administrators, either by persons who desire to recommend the candidate or by the candidate himself, not later than 31st December, 1960, by letter addressed to The Convenor of the Administrators, Sir George Beilby Memorial Fund, The Royal Institute of Chemistry, 30 Russell Square, London, W.C.1. The letter should be accompanied by nine copies of a short statement on the candidate's career (date of birth, education and experience, degrees and other qualifications, special awards, etc., with dates) and of a list of titles with references, of papers or other works published by the candidate, independently or jointly. Photographic copies of these documents are acceptable. Candidates are also advised to forward one reprint of each published paper of which copies are available.

## Dislocation Theory Course

THE contribution of dislocations and point defects to the physical and mechanical properties of crystalline solids, particularly metals, cannot be over-emphasised, and a knowledge of dislocation theory is an essential tool for all those who seek to understand these properties. Organised by the Extra-Mural Department of the University of Liverpool a course of ten lectures on the subject will be given by Dr. D. Hull during the Autumn Term.

The course is designed for physicists and metallurgists with little or no preliminary knowledge of dislocation theory. The concept of a dislocation will be described and then a detailed account of the properties of dislocations will be given. The course will include demonstrations of the methods used for observing dislocations, and in particular, direct observation in thin metal film in the electron microscope. The application of the theory will be illustrated by examples from the mechanical properties of metals. The course will include the following topics: (1) definition of dislocation, (2) elastic theory of dislocations, (3) movement of dislocations, (4) dislocation interactions, (5) partial dislocations and stacking faults, (6) crystal growth, (7) recovery and recrystallisation, and (8) application of dislocation theory to work hardening, fatigue, creep and precipitation hardening. The lectures will be given in the Department of Metallurgy, University Main Building, Brownlow Hill, Liverpool, on Monday evenings, commencing 10th October, 1960. The fee for the course is £2 12s. 6d.

## Engineers at Scunthorpe

THE Iron and Steel Engineers Group of The Iron and Steel Institute has chosen Scunthorpe as the location for its forty-first meeting, from Monday to Thursday, October 24th-27th, 1960. The programme will include technical sessions at which papers from local authors will be presented and discussed. E. A. Atkin and E. F. Farrington have contributed a paper on developments at Appleby-Frodingham since 1954, and D. R. M.



Nisbet of the same company has written a study of lighting in the iron and steel industry. From the Redbourn section of Richard Thomas and Baldwins, Ltd., comes a paper on engineering problems in re-developing an integrated iron and steelworks, by P. M. Hesketh, whilst F. E. Stacey of John Lysaght's Scunthorpe Works, Ltd., is the author of a paper on the investigation of failures in a blooming mill. In addition to the technical sessions, to be held at Scunthorpe's Civic Theatre, there will be a series of visits to the three iron and steelworks in the area, Appleby-Frodingham, Redbourn, and Lysaght's. The Lincolnshire Ironmasters' Association will be hosts to those attending at a dinner at the Wortley Hotel, Scunthorpe, on the evening of October 26th. It is expected that about 150 members of the group from all parts of the country and representing many branches of engineering will be attending the meeting.

### **"Stone into Steel" Wins Premier Award**

THE United Steel Cos., Ltd., have gained the premier award at the Venice Film Festival with their film "Stone into Steel" out of a total entry of 240 films from all over the world. Featuring the works of Appleby-Frodingham Steel Company, this colour documentary has no spoken commentary but the dramatic effect is heightened by a specially-composed musical score. It was shown to United Steel's shareholders at a series of special performances in cinemas all round the country during June and July. The Golden Mercury trophy was presented to the producer, Mr. A. Frank Bundy of Wallace Productions by the President of the Venice Chamber of Commerce on July 30th. Copies of "Stone into Steel" in 16mm. and 35mm. size are available on free loan on application to the public relations department of United Steel.

### **Castner Memorial Lecture**

DR. W. J. KROLL of Corvallis, Oregon, U.S.A., will deliver the Sixth Castner Memorial Lecture of the Society of Chemical Industry under the title "The Fusion Electrolysis of Titanium" on Friday, October 7th, 1960 at the Royal College of Science and Technology, Glasgow, C.I. (Room 24) at 6 p.m. All interested persons will be welcome.

### **Symposium on Electrical Contacts**

THE Institute of Physics and The Physical Society announces that it is arranging a Symposium on Electrical Contacts in collaboration with The Institution of Electrical Engineers which will take place in Brunel College of Technology, London, on 5th-7th April 1961. The symposium is intended to cover recent advances in the study of the phenomena occurring at mating surfaces carrying currents used in light electrical engineering. It will include electrical erosion and material transfer, mechanical wear, and the influence of surface films and contamination, and will deal with make-and-break contacts, sliding contacts, semi-permanent contacts and connections, between metallic and non-metallic materials. In view of the nature of current developments, it is considered that major interest will be focussed on aspects relating to contact resistance. A number of short papers have already been invited by the committee which will

be glad to consider offers of other contributions. These should be submitted by 30th September next and be accompanied by a brief outline of the proposed contributions (50-100 words). All communications regarding the symposium should be sent to the Secretary, The Institute of Physics and The Physical Society, 47 Belgrave Square, London, S.W.1.

### **Film Guide**

THE August issue of *Film User* contains that journal's annual survey of informational films. The survey is in fact divided into two parts, and that contained in the August issue takes the form of an alphabetical directory of firms and organisations from which 16 mm. prints can be borrowed or hired, showing their addresses and conditions of loan, and outlining the topics covered by their films. The September issue will contain a classified index of subjects under more than two hundred different headings. Single copies may be obtained (price 2s., post paid) from the publishers, Current Affairs, Ltd., 319 High Holborn, London, W.C.1.

### **"When It's Really Hot"**

IN recent years Henry Wiggin & Co., Ltd., has organised fifteen exhibitions in the main industrial centres of the country, but the latest—"When It's Really Hot"—is the first to concentrate exclusively on Wiggin high-temperature alloys. Opening at the Grand Hotel, Sheffield, from September 20th to 22nd, and continuing at More's Hotel, Glasgow, from September 27th to 29th, it will include the Nimonic series, the Nimocast series, Inconel, Incoloy and Incoloy DS, and will demonstrate properties and applications of these alloys by diagrams and animations and a comprehensive range of exhibits of components and sub-assemblies in which Wiggin materials are used. With the growing importance of these alloys to industry and the proven applications in the aircraft world, the exhibition should provide an interesting technical story for engineers and designers. The exhibition will be supplemented by lectures on high-temperature problems given by the company's technical service staff and supported by film showings.

### **Galvanising Conference**

THE sixth in the series of International Conferences on Hot Dip Galvanising is to be held in Switzerland—at is the Kursaal, Interlaken—from 4th-9th June, 1961, and not in France as previously announced. The number of delegates attending the previous conferences at Copenhagen, Düsseldorf, Oxford and Milan has steadily increased, and nearly 350 were present at the 1958 Conference held in Holland and Belgium in conjunction with the Brussels World Exhibition. Numbers at Interlaken are expected to be even bigger.

Three days will be devoted to technical sessions at which more than twenty papers will be presented on works practice, the properties and corrosion resistance of galvanised coatings, the welding of galvanised steel, the metallurgy of galvanising, after-treatments, and the heating of galvanising baths. One session will also be devoted to sheet galvanising. Preprints of the papers will be issued in French, German, Italian and English, and there will be simultaneous translation into the same four languages at the technical sessions themselves. The edited proceedings of the conference, to be pub-



lished towards the end of 1961, will be issued free to all delegates, and should take their place alongside the proceedings of earlier conferences as part of the authoritative literature on hot dip galvanising.

Works visits will be arranged in conjunction with the conference to general galvanising plants in Switzerland, and to sheet, wire and tube galvanising plants in Italy. Plant, equipment and materials supplied to the galvanising industry will be exhibited by manufacturers in a room adjacent to the conference hall.

Those wishing to attend the conference as delegates or to participate in the exhibition of plant, equipment and materials should write to the Zinc Development Association, 34 Berkeley Square, London, W.1.

### Robert Horne Memorial Lecture

THE Fourth Robert Horne Memorial Lecture of the Society of Chemical Industry will be by Professor F. D. Richardson, entitled "The Extraction of Metals and the Chemistry of Metals" and will be delivered in the lecture theatre of the Chemistry Department of the University of Bristol, Woodland Road, Bristol 8, at 6.30 p.m. on Thursday, September 29th, 1960. All interested persons are welcome to attend.

### Automatic Control Symposium

MEMBERS of the constituent societies of the British Conference on Automation and Computation will be aware that the First International Congress of the International Federation of Automatic Control (I.F.A.C.) was held in Moscow from 27th June to 7th July this year. The Congress was international and the British contribution consisted of twenty-nine papers. Arrangements are now being made by the Institution of Mechanical Engineers for discussion of the British papers at a symposium in London, on 27th and 28th September 1960, for the benefit of those who are interested in this important subject, but were not able to attend the Moscow Congress. In addition to the discussion of the papers it is planned to report briefly the more important contributions to the Moscow Congress. Preprints of the British papers are available and will be supplied to those registered to attend the symposium. A list of the papers, together with a registration form to attend the symposium, may be obtained on application to the Secretary of the Institution of Mechanical Engineers, 1 Birdcage Walk, Westminster, London S.W.1.

### Steelworks Automation Unit

AN important step towards meeting rapidly increasing requirements for automation in the steel and allied industries has been taken by Associated Electrical Industries, Ltd., and Davy-United, Ltd., who have set up a jointly-financed steelworks automation unit to apply new automatic control techniques to the processing of both steel and non-ferrous metals. The problems involved in full automation in the steel and allied industries are, however, too complex for any single existing manufacturing organisation to deal with adequately within its own resources. A.E.I. has therefore allied with Davy-United to establish this combined unit, which has been specifically organised to deal with such problems from the stage of the initial design study to the commissioning of a fully-operational production system in a plant.

The combination of A.E.I.'s long experience in the development of electronic and electrical equipment with that of Davy-United in building steelworks plant and developing specialised control for steelworks, backed by the large research resources of the two companies, will enable the new unit to undertake comprehensive automation projects which can be expected to offer considerable long-term benefits in improved productivity and economy of operation. Such projects will include the combination of advanced servo controls with computers to ensure optimum performance of rolling mills and associated plant.

The A.E.I./Davy-United Steelworks Automation Unit has its headquarters at Mill Road, Rugby, with representatives at both Davy-United, Ltd., Sheffield and A.E.I. Heavy Plant Division, Rugby.

### Coking Plant Film

THE film of the Merton coking plant shown recently is a joint production of the Durham Division, National Coal Board and Woodall-Duckham Construction Co., Ltd. The filming of the plant took place during 1959, and for the greater part during the normal operation of the plant. Of special interest are the sequences of the Wingfield beehive ovens, the last plant of this type in operation in Europe. These were shot before the plant closed down in the autumn of 1958.

The film is a 16 mm. colour sound film, with single perforation and full width optical sound track, 900 ft. in length, with a running time of approximately 25 minutes: the projection speed is 24 frames per second. The film was produced and photographed by Mr. W. A. Speed, publicity manager of Woodall-Duckham Construction Co., Ltd., in conjunction with Turner's Film Unit of Newcastle. The commentary was read by Mr. Kenneth Kendall and the introduction on the film was spoken by Mr. M. D. Edington, carbonisation director of the Durham Division of the National Coal Board.

### New Vacuum Engineering Company

A STATEMENT by Elliott-Automation, Ltd., announces that a joint company, Leybold-Elliott, Ltd., is being formed by Elliott Brothers (London), Ltd., and Leybold's of Cologne. The new company will exploit throughout the British Commonwealth, except Canada, the industrial vacuum process equipment of both its parent groups, and by combining the complementary knowledge and experience of Leybold and Elliott will expand the range of products still further. By a reciprocal agreement these products and those of Elliotts may be marketed in certain countries in Europe by the Leybold companies. E. Leybold's Nachfolger of Cologne manufactures an extensive range of vacuum components which include high efficiency rotary and diffusion pumps, gauges and other items of equipment used in high vacuum industrial processes; its associated company, Leybold Hochvakuum-Anlagen G.m.b.H. has specialised in the design and supply of advanced systems based upon high vacuum techniques. The Elliott Vacuum Physics Research Laboratory at Borehamwood has for a number of years been working in specialised fields of high vacuum physics, and has developed a range of very advanced techniques which have been applied principally in developing high power electronic radar equipment and in specialised leak-detection installations for inspection, health monitoring and quality control.

Leybold-Elliott, Ltd., is holding a three day exhibition of its products at the end of September in the London area at which the applications of high vacuum techniques will be discussed with representatives of many fields of industry. Invitations to the exhibition can be obtained from Leybold-Elliott, Ltd., c/o Elliott Brothers (London), Ltd., Elstree Way, Borehamwood, Herts.

### Non-Destructive Testing Course

FOLLOWING the success of the course on "The Techniques of Non-Destructive Testing" held last year, a further course of ten lectures entitled "Advances in Non-Destructive Testing" will be held in the Physics Department of the Brunel College of Technology, Woodlands Avenue, Acton, London, W.3. The lectures, given by well-known specialists in the subject, will be held on Wednesday evenings from 7 to 8.30 p.m. commencing on 28th September, 1960. They will deal with latest developments in the subject and should appeal not only to graduates and holders of the Higher National Certificate in engineering and science subjects but also to inspectors and others who have an interest in non-destructive testing. The fee for the course is £1 and further details may be obtained from the Head of the Physics Department.

### Metal Cleaning, Ltd.

METAL CLEANING, LTD., is the new name given to the recently acquired subsidiary of the Castrol group. The previous title of De-Corrosion Services (Norwest), Ltd., has been discontinued. The company provides services to clean corroded metals and remove all other forms of metal contamination, the techniques used including electro-chemical and chemical immersion, and flame and shot blasting. Once the essential task of cleaning is completed, appropriate protective treatments are applied.

The processes can be carried out either on the spot or at the company's Liverpool works, 2-6 St. John's Road, Bootle 20. It is planned to acquire a new factory in the Liverpool area which will have a capacity at least four times as great as the present plant and will be the main centre for service, development and research. Metal Cleaning, Ltd., also intend to establish a chain of depots throughout the country in the major marine and industrial centres.

### Austenite Transformation Course

A VACATION COURSE on The Transformation of Austenite is to be held in the Metallurgy Department of the Battersea College of Technology from 27th to 30th September, 1960. The course has been designed to show theoretical advances in a subject that has great historical interest and is of fundamental commercial importance. Lectures will cover the following topics: the thermodynamics and kinetics of transformation, the effect of alloying elements on the stability of austenite, isothermal and continuous cooling, the mechanical properties of steel, the austenitic steels, the pearlite reaction, the bainite reaction, the martensite reaction, and the tempering of martensite: ample time will be allowed for discussion of each topic.

The fee for the course is 12 guineas, inclusive of luncheon and morning and afternoon refreshment. Enrolment forms may be obtained from the Secretary (Metallurgy Courses), Battersea College of Technology, London, S.W.11.

## News in Brief

CONTRACTS for rolling-mill drives valued at nearly half a million pounds and totalling 14,000 h.p. have been obtained from steel companies by the Heavy Plant Division of Associated Electrical Industries, Ltd. Motors accounting for half this horsepower will be installed in the Stocksbridge (Sheffield) works of Samuel Fox and Co. Ltd. The other orders are from the Head Wrightson Machine Co., Ltd. (for installation at Tubes, Ltd.) and the South Durham Steel and Iron Co. Ltd.

At a recent extraordinary general meeting of C. C. Wakefield & Co., Ltd., a decision was taken to change the name to Castrol, Ltd. The company was founded in 1899 by Charles Cheers Wakefield, later Viscount Wakefield of Hythe, and Castrol motor oil made its appearance ten years later.

THE WEAN ENGINEERING Co., of Warren, Ohio, has established a new company in the U.K.—Wean-Miles, Ltd.—to furnish strip and tinplate mill equipment of Wean design. The new company will assume the manufacture and sale in the U.K. of many items of equipment designed and sold by Wean in the U.S.A. and throughout the world. The head office of Wean-Miles, Ltd., is at 76 Cannon Street, London, E.C.4.

ALLEN AND MACLELLAN (POLMEDIE), LTD., a subsidiary of Glenfield and Kennedy Holdings, Ltd., has arranged on suitable financial terms to transfer its compressor business at Polmedie (excluding fixed assets) to G. & J. Weir. The fixed assets—land, building and plant—at Polmedie have been purchased by Davy and United Engineering Co., Ltd., to enlarge their productive capacity in Scotland.

AN order worth about £700,000 for electrical equipment for a new rod mill has been obtained by the Heavy Plant Division of Associated Electrical Industries, Ltd., from the Broken Hill Pty. Co., Ltd., Australia. Most of the electric control equipment required will be manufactured in Australia by Australian Electrical Industries (Pty.), Ltd. Responsibility for manufacturing the rest of the equipment will be divided between the Rugby and Manchester (Trafford Park) works of A.E.I.

THE GEIGY Co., LTD., of Rhodes, Middleton, Manchester, has acquired subject to contract the offices and laboratories of the British Rayon Research Association at Heald Green, Manchester. The site and buildings have become available since the Association, as already announced, is to merge with the British Cotton Industry Research Association (Shirley Institute) at Didsbury. The intention is to transfer the offices of Geigy (Holdings), Ltd., and the essential headquarters of the Geigy Company's sales, administration and laboratory activities from Rhodes to Heald Green some time during 1961.

AN order worth about £500,000 for rectifiers and transformers for Hindustan Aluminium Corporation has been awarded to The English Electric Co., Ltd. The equipment, to be shipped to a new plant in the spring of 1961, will supply direct current power for aluminium smelting. The rectifiers and transformers in this order will be made at the Stafford works of English Electric.

AN agreement has been made between the Palmer-Bee Co. of Detroit, Michigan, U.S.A. and Brown, Lenox & Co. Ltd., of Pontypridd, Glamorgan, South Wales, for the manufacture in this country of Palmer-Bee heavy duty conveyors and auxiliary rolling mill equipment.

# RECENT DEVELOPMENTS

## MATERIALS : PROCESSES : EQUIPMENT

### Pocket Graticule and Magnifier

A POCKET magnifier with built-in graticule that folds into a neat square measuring only  $2\frac{1}{8}$  by  $1\frac{7}{16}$  by  $\frac{9}{16}$  in., and can be comfortably carried in the waistcoat or breast pocket has recently been introduced by Graticules, Ltd. It has been designed, amongst other requirements, to fill the gap between rough estimation with a scale and precise measurement with elaborate and expensive instruments, and will no doubt find application in many fields, graticule patterns being available to meet individual requirements.



As will be seen from the illustration, the micrometer, which is on glass, is mounted in the base of a low powered folding magnifier, the whole frame being made of aluminium, cut away to give maximum visibility, the field of view being 1 in. square. The micrometer scale is placed in close contact with the object to be measured or viewed and the scale shows up well against a wide variety of colours, whilst the graduations are sufficiently fine to ensure a reading accuracy of  $\pm 0.002$  in. Graticules, Ltd., in the production of their scales, recently evolved what they call their maXta process, which gives an ideal surface image firmly bonded to the glass and resistant to heat, chemical attack and abrasion to the same extent as the glass itself.

*Graticules, Ltd., Bath House, 57/60 Holborn Viaduct, London, E.C.1.*

### Protective Paper Tape

A LOW adhesion paper tape for protection of polished stainless steel or other highly finished surfaces during fabrication or in transit. Permacel 95 protective paper tape is a thin yet exceptionally strong paper tape with a low tack, non staining, pressure sensitive adhesive designed specifically for protecting various types of polished surface. Among the claims made for this product are: that it gives maximum protection to stainless steel, aluminium, or plated components during forming, bending, fabrication and shipment; and that it

is resistant to abrasion and gives adequate adhesion to even the most highly polished surface, yet when removal is desired strips cleanly.

*Permacel Tapes Ltd., 260 Bath Road, Slough, Bucks.*

### High Speed Cutting Nozzles

BRITISH OXYGEN GASES, LTD., have developed a range of oxy-acetylene nozzles which give faster machine cutting speeds than any existing models. These new high speed nozzles have been developed for machine cutting applications and are an addition to the "Saffire" range of equipment. They can cut 7 in. thick plate at 45 ft./hr. and 1 in. plate at 100 ft./hr. They are available in four sizes: 45, 50, 60 and 73. the size numbers indicating the diameter of the throat passages in thousandths of an inch.

The new nozzles will lower the production costs of machine cutting and reduce the time spent on finishing and machine work. They are of one-piece construction and, apart from the conical seating surface, are chromium plated. The cutting orifices, which are machined to exacting limitations, are of convergent-divergent form. This allows the cutting oxygen supply to expand to atmospheric pressure within the nozzle. The cutting oxygen leaves the nozzle at high speed in a parallel stream, resulting in an exceptionally high linear cutting speed.

*British Oxygen Gases, Ltd., Spencer House, St. James's, London, S.W.1.*

### Heavy Duty Gas Pressure Switches

NEW gas pressure switches have been introduced as part of their range of simple non-indicating industrial controllers by Honeywell Controls, Ltd. Designed for heavy duty under arduous operating conditions, these





diaphragm-actuated mercury switch controllers can be used as safety cut-off switches or simple pressure controllers for both hazardous and non-hazardous gases.

They feature a seal-off diaphragm which prevents gas from entering the switch chamber should the main diaphragm be ruptured. This, together with the high overload capacity, confers a high degree of safety operation. With non-hazardous gases, the switches can be used as differential pressure controllers since the seal-off chamber becomes the low pressure side.

Pressure ranges from  $\frac{1}{2}$  in. W.G. to 5 in. W.G. to 10-100 lb./sq. in. are available. Switching action is SPST, make or break on pressure rise according to model: SPDT versions are available. A lockout feature, optional on some models, standard on others, prevents re-cycling when the pressure returns to preset limits, having once broken circuit. This feature can be adjusted to eliminate the lockout if desired. Specification S1011-1a giving full details is freely available.

*Honeywell Controls, Ltd., Ruislip Road East, Greenford, Middlesex.*

### Melting and Holding Furnace

AN improved type of aluminium melting and holding furnace which, unlike conventional models, has no crucible, is designed to permit replacement of elements without loss of production. It can be used as a holding or a melting furnace, or as a combination of both, and is suitable for many types of metal including aluminium and zinc alloys. Heating is by means of silicon carbide rod type elements which radiate down on to the metal and are controlled to prevent their overheating and ensure long life. The whole furnace is lined with a suitable iron-free refractory with a liberal insulation backing.

In operation standard billets are fed into the bath and as the aluminium melts further ingots can be added until the aluminium is within 3 in. of the top of the bath; baling out takes place through the bale-out section. Thermostatic control maintains the metal

at the correct temperature to give a product of high purity and minimise metal loss through oxidation and volatilisation which has been found to be about 1%. The furnace is rated to provide 5 lb. of molten aluminium per KWh., melt and hold.

*Hedin, Ltd., Commerce Estate, South Woodford, London, E.18.*

### Surface Treatment of Aluminium

A NEW range of treatments for aluminium alloys, comprising Bonderite 710, Bonderite 701, Bonderite 740 and Aluma Etch 391 has been introduced by the Metal Finishing Division of Pyrene, Ltd. The Bonderizing techniques represent an extremely economical low temperature method of coating aluminium and convert the aluminium surface into a thin amorphous oxide-chromate coating which is integral with the metal itself.

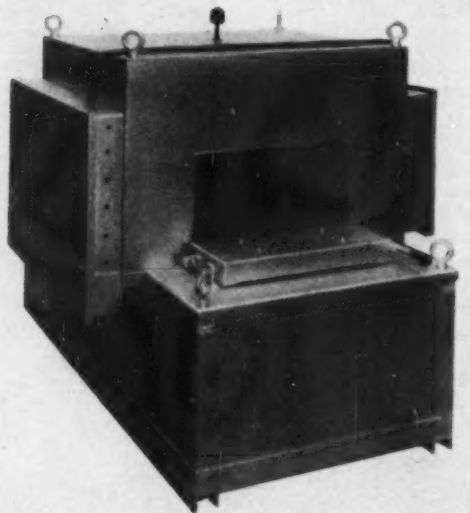
Bonderite 710 coatings are of an attractive well-defined golden colour, though variations in tone may occur according to the different alloys used. The applications and potentialities of this process are extremely diverse. It will treat with equal efficiency such varied subjects as small aluminium parts and continuous strip. Bonderizing is in use on strip lines in this country for the treatment of aluminium strip at over 60 ft./min. Processing times are exceptionally low, offering fast and economic production techniques. The amorphous Bonderite coating, itself inseparable from the metal surface, tightly bonds the paint so that amazingly complex forming operations can be performed without impairing paint adhesion.

The Bonderite 701 process converts the aluminium surface into an amorphous oxide-chromate coating of a pleasant green colour which is integral with the metal and is light-fast. The coating, in its own right, offers excellent corrosion protection to aluminium and aluminium alloys. Bonderite 701 has many architectural applications where durability and colour count.

Bonderite 740 is another oxide-chromate treatment providing a coating which is both an ideal base for organic finishes, and a corrosion resistant film in its own right. It can be applied by immersion, spray or in a continuous strip line. It has been specially developed for treating aluminium and aluminium alloys in mixed production with steel.

Aluma Etch 391 is a scale free, controlled alkaline aluminium etchant for uniformly etching the surface of aluminium in an immersion bath. Unlike other etching solutions where dissolved aluminium reacts with caustic soda to form insoluble sodium aluminate, the special formulation of Aluma Etch prevents the conversion of sodium aluminate into sludge. The use of Aluma Etch therefore produces no suspension and no precipitate. Moreover, once the desired degree of etch has been established for a particular alloy, operations may be easily controlled to produce the required etch, uniform and evenly diffused, no matter what the size of surface area to be covered. Aluma Etch 391 is applied by immersion and the parts to be treated are immersed in the hot solution for a predetermined time to remove soil and to produce the required degree of etch. On removal from the bath, the parts are merely cold water rinsed in a bath sufficiently overflowed to prevent contamination.

*The Pyrene Co., Ltd., Metal Finishing Division, Great West Road, Brentford, Middlesex.*





# CURRENT LITERATURE

## Book Notices

### A SYMPOSIUM ON ALUMINIUM IN BUILDING

Proceedings at Symposium on Aluminium in Building held at The Royal Institute of British Architects in London on July 9th and 10th, 1959. 256 pp. 4to., soft covers, numerous illustrations and sketches. London, 1960, The Aluminium Development Association, 33 Grosvenor Street, W.1. 50s.

THE uses of aluminium for building have received considerable impetus over the past few years from developments initiated, for the most part, in the aluminium industry and allied trades. New aluminium forms, such as larger extruded sections, longer and wider sheets; improvements in joining and finishing techniques; and researches directed towards greater efficiency in design—all these have widened the scope for architect and designer.

A Symposium organised by the Aluminium Development Association, and held in July of last year at The Royal Institute of British Architects, provided a timely opportunity for exchange of ideas and information between architects, builders, engineers, and associated manufacturers on the one hand, and on the other, the aluminium industry, the principal objective being to receive suggestions and criticisms which should help forward the development of further new items by the aluminium industry and manufacturers.

The report of the symposium falls naturally into three divisions, dealing with aluminium as a building material (3 papers); aluminium curtain walling (4 papers); and aluminium building applications (6 papers). The second session included surveys of curtain walling in this country, in North America, and in Europe, and the first of these papers gives section and assembly drawings of sixteen British systems, with photographs of typical installations. Other papers deal with economic aspects; standards and codes of practice; finishes; roofing and vertical cladding; windows; furniture; and with pre-fabricated and transportable buildings; also, in two papers, with research into problems of thermal insulation, and aluminium in relation to the Thermal Insulation Act. The discussion ranged from the uses of foil to construction of big domes, from the fire hazard to the supply of aluminium nails, and from mastics to maintenance, and its value as discussion was the greater for the presence, and the contributions, of the representatives of no less than twelve nations; from their diverse viewpoints emerged many indications of future possibilities for extending the usefulness of aluminium in this important field of application.

The present volume reproduces the papers in full, together with both oral and written contributions to the discussion.

### PROGRESS IN METALLURGICAL TECHNOLOGY

147 pp. including 37 diagrams in the text and 16 pp. of art plates. London, 1960, Iliffe & Sons, Ltd. (for the Institution of Metallurgists). 32s. 6d. net (by post 33s. 4d.)

EVERY year since 1947 the Institution of Metallurgists has held a Refresher Course for its members, during which leading authorities survey the present state of knowledge in relation to a particular aspect of metallurgy.

Originally these papers were republished in book form, solely for the benefit of Institution members, but the information they contained was considered far too valuable for only a limited circulation. Therefore, in conjunction with the present publishers, it was arranged that all papers read at Refresher Courses from 1956 onwards should be made available to a wider public. The response to the publication of the first three volumes under this scheme was very gratifying, and it is felt that the demand for this fourth volume will certainly be at least as satisfactory.

At the 1959 Refresher Course the present position of metallurgical technology was reviewed and recent developments and trends discussed. In the first paper in this volume, Dr. John Taylor deals with modern developments in the production of iron and steel, including the revolution which is now taking place in steel-making practice due to the advent of so-called tonnage oxygen. Mr. Bradshaw, in the paper which follows, outlines the various methods now in use for the extraction and refining of non-ferrous metals, and gives a detailed description of the newer processes. Amongst other things he deals with autogenous grinding, hydro-cyclone classification and fluidised bed roasting. He also discusses hydro-metallurgical techniques for the exploitation of low grade ores and the influence of ion exchange and solvent extraction processes. A further section covers developments in pyrometallurgy.

In the third paper Dr. Robiette deals with the melting of ferrous and non-ferrous metals, and shows that although methods have not changed greatly throughout the years, the introduction of improved equipment and greater mechanisation has led to increased efficiency and the production of purer metals. Finally, Dr. D. V. Atterton, in his paper on casting, shows how it has been possible to attain at the same time both an increased rate of production and a high level of quality.

## Trade Publications

WE have received from S. Russell & Sons, Ltd., Leicester, copies of three new leaflets dealing with the company's alloy iron production. The first gives details of the properties of Spheronic, which is the registered trade name of Russell's austenitic heat resisting cast iron with spheroidal graphite. A second publication is concerned with Ni-Resist austenitic cast iron in both the flake graphite and spheroidal graphite types. The last leaflet illustrates the use of S.G. iron at the top of the 450 ft. chimneys of the High Marnham power station, and gives details of S.G. iron specifications.

A NEW, larger edition of Keith Blackman's "Heavy Fan Engineering" booklet (Publication No. 25) has now been published and is available from the Publicity Dept., Mill Mead Road, London, N.17. This fully illustrated booklet is designed to show the scope of the "Tornado" range of fan engineering equipment in the large and heavy sizes. Individual sections deal with centrifugal and axial fans, blowers and exhausters, ancillary equipment such as air heaters, dust exhaust and collecting plant, and metal structure and fabrications.

IMPALCO C80 "SUPERSPEED" is a medium strength heat-treatable aluminium alloy containing small additions of lead and bismuth evenly dispersed as fine particles throughout the material. These act as chip breakers and allow high speed production of machined parts with a good surface finish. A leaflet issued by Imperial Aluminium Co., Ltd., a subsidiary of Imperial Chemical Industries, Ltd., describes this new material.

NUMBER 3 of a series of sales information bulletins issued by the Metal Finishing Division of The Pyrene Co., Ltd., describes Pyroclean No. 9, a heavy duty general purpose degreasing agent of the alkali type, which is suitable for both still plants and spray machines.

We have received from Thompson L'Hospied and Co., Ltd., a copy of a new leaflet, No. V.41B, which gives details of their cast nickel chromium carburising boxes, which may be circular or rectangular in section.

"MILLING CUTTERS" AND "REAMERS" are the respective titles of Nos. 12 and 14 in the series of brochures dealing with tools manufactured by Samuel Osborn & Co., Ltd., of Sheffield. Details are given in each case of the individual tools covered by the Osborn range.

STILLITE PRODUCTS, LTD., have recently issued a revised publication (Technical Data No. 5/60) dealing with the use of mineral wool slabs in place of Diatomite as a backing to refractory linings for furnace and stove insulation. The same quality is also available in the form of Stilag pipe sections, which are covered by publication Technical Data No. 7/60.

OF two publications received from George Kent, Ltd., No. 281 gives particulars of the standard orifice fittings developed by Kent over the last fifty years, covering normal flow requirements for steam, air, gas or liquid measurement. The other publication, No. 109, is concerned with the J.S.M. domestic water meter.

THE INCANDESCENT HEAT COMPANY's leaflet, V.61, deals with the radiant-jet batch furnaces made by the company for general and controlled atmosphere heat treatments within the temperature range 700-1,000° C. Standard size work chamber dimensions are 3 ft. long by 2 ft. wide by 1 ft. high; 4 ft. long by 3 ft. wide by 1½ ft. high.

THE Liverpool works of the English Electric group of companies has a specialised foundry for the production of castings of the gravity, pressure die and shell moulded types. This foundry is in a position to undertake work from outside customers, and a publication illustrating the facilities available can be obtained from The English Electric Co., Ltd., Die Casting Department, East Lancashire Road, Liverpool, 10.

THE Spring issue of *This is Magnesium* contains features dealing with pressure diecasting, magnesium-plastic laminated suitcases, and the use of magnesium in stratospheric flights and deep sea diving.

THE INCANDESCENT JETUBE, originally developed for gas carburising furnaces, has found widespread application in radiant tube heating practice. A leaflet recently issued by The Incandescent Heat Company, Ltd., deals with their use in vitreous enamelling furnaces of both batch and continuous types.

*Kodak Professional News* is the title of a new quarterly publication whereby Kodak, Ltd., hope to provide a more up-to-date and comprehensive information service than has been possible through existing channels. Many of the items contained in the first issue are the result of enquiries received from customers on such subjects as

retouching Ektacolor prints, close-up lenses for use with Retina cameras, and the checking of safelights.

THE April issue of *Foundry Developments*, issued by Foundry Services International, Ltd., includes articles on hydrogen in steel, degassing copper base alloys, the effect of degassing treatment on the sodium content of aluminium-silicon alloys, and the refinement of hypereutectic aluminium-silicon alloys.

THE Alloys Division of Union Carbide, Ltd., is at present engaged in revising its *Ferro Alloys and Electro Metallurgical Products' Catalogue*. This will take some time and in the meantime the Division has issued a buyers' easy reference catalogue of 150 ferro alloys and other materials.

THE popular and useful Copper Development Association publication *Copper Data* has now been revised in conformation with the standardised format adopted for all the many other C.D.A. technical publications. Throughout some twenty-five years of publication, *Copper Data*, with its general but comprehensive information on the properties, treatments and working of copper, together with details of commercial grades and applications, has served as an introductory guide to engineers, technicians, students and all concerned with the use of this metal.

THE June issue of *Close-Up*, the house journal of Armstrong Whitworth (Metal Industries), Ltd., and Jarrow Metal Industries, Ltd., contains an article on iron rolls, the third of a series on roll types and applications. A further technical article describes the development of electric furnaces from the early days, when the capacity was of the order of a ton, to the present day, when furnaces of 150 tons capacity are in service.

ORGANIC CHEMICAL REAGENTS MONOGRAPH No. 39 (1960), issued by Hopkin & Williams, Ltd., of Chadwell Heath, Essex, deals with xylenol orange, one of a series of metal indicators obtained by the Mannich condensation of a sulphonphthalein indicator with formaldehyde and iminodiacetic acid. Details of its use in the determination of zinc, lead, mercury and bismuth are described fully and the modifications in detail required for other metals are referred to briefly.

## Books Received

"Design and Performance of Gas Turbine Power Plants." Editors: W. R. Hawthorne and W. T. Olson. Vol. XI, "High Speed Aerodynamics and Jet Propulsion." 563 pp. inc. index. Princeton and London, 1960. Princeton University Press and Oxford University Press. 105s. net. in U.K. only.

"Surface Effects on Spacecraft Materials." Transactions of the symposium co-sponsored by the Missiles and Space Division of Lockheed Aircraft Corporation and the Air Research and Development Command of the U.S. Air Force, Palo Alto, California, May, 1959. Edited by F. J. Clauss. 404 pp. New York and London, 1960. John Wiley & Sons, Inc., and Chapman & Hall, Ltd. 92s. net.

"Spectrochemical Abstracts." Vol. VI. 1954-1955. By E. H. S. van Someren and F. Lachman. 100 pp. inc. author and elements indexes. London, 1960. Hilger & Watts, Ltd. 25s.

"Methods in Geochemistry." Edited by A. A. Smales and L. R. Wager. 464 pp. inc. subject and author indexes. New York and London, 1960. Interscience Publishers, Ltd. \$13.50 or 94s.



During wartime, gas masks were issued for filtering out poisonous and harmful constituents from air in the event of a gas attack. Birlec adsorption dryers are used for removing water vapour from process gases employed in modern manufacturing techniques. For process protection in the metallurgical, chemical and electrical industries, dew-points below  $-40^{\circ}\text{C}$ . are frequently required. Standard Birlec dryers are available for such applications and specialised equipment can be built for obtaining dew-points down to  $-100^{\circ}\text{C}$ . Users of one or more of the gases listed alongside are invited to write to Birlec for a copy of Publication No. 82/4.



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## Dielectric Heating - 3

Some further details of the uses to which dielectric heating can be put are given in this data sheet, being continued from data sheet No. 11.

### The Woodworking Industry

A most important development in recent years in the woodworking industry has been the introduction of synthetic resin adhesives of the thermosetting type for the bonding and adhesion of wooden components.

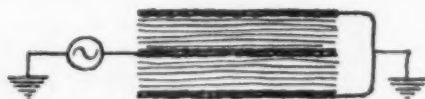
Setting of these resin adhesives proceeds at a rate largely determined by temperature. For instance, urea formaldehyde, one of the resins in common use, sets as follows:

TEMPERATURE	SETTING TIME
65°F	3 hours
80°F	1 hour
150°F	3 minutes
190°F	1 minute

The resultant bonded joint is equally satisfactory in each case. Most of the power supplied when dielectric heating is used is absorbed by the resin, the heat thus being concentrated where required and power consumption reduced to a minimum.

### Plywood

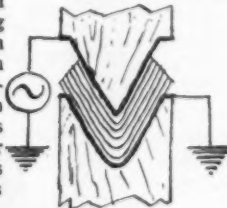
With dielectric heating consuming power only during the heating cycle, plywood can be produced with considerable savings in heating times and costs.



For example, in a press holding 100 3-ply  $\frac{1}{8}$ " thick assemblies, the resin glue is set in 20 to 30 minutes, depending upon the dryness of the wood. An output of up to 60 cu. ft. of plywood is obtained per hour using a 25 kW H.F. generator.

### Curved Laminated Sections

Curved laminated sections are being increasingly used in contemporary furniture, and with dielectric heating rapid production can be achieved using wooden shaping blocks in single daylight presses. An alternative method of providing heat by conduction from heated metal strips becomes increasingly slower as the total section thickness rises above 0.05 inch, as shown below:

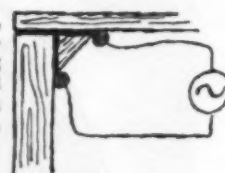


TOTAL THICKNESS OF LAMINATIONS	Comparative heating times in minutes	
	CONDUCTION	DIELECTRIC
1 inch	20	4
$\frac{1}{2}$ inch	5 $\frac{1}{2}$	2 $\frac{1}{2}$
0.6 mm veneer	1	1 $\frac{1}{4}$

### Furniture Assembly

Because of the savings in gluing processes already instanced, dielectric heating is being extensively used in the furniture trade. It leads also to reductions

in labour and floor space, with the elimination of assembly jigs. The heating equipment can be placed directly in the production line, cutting handling to a minimum.



### Resin-bonded Wood Chipboard

A substitute for natural timber is made from wood waste and chippings, broken down to a coarse size, mixed with synthetic resin and heated under pressure. Dielectric heating gives quick and uniform heating, and increased fluidity reduces the power required for the final pressing and curing operation. In a continuous process, the length of the press required is also reduced.

### Blockboard

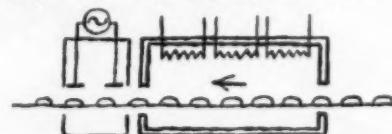
Production of blockboard by edge gluing strips of wood also provides an excellent use for dielectric heating, since considerable savings in time and labour can be effected owing to the large areas of glue line involved.

### Other Resin-bonded Products

Dielectric heating is also used in the manufacture of other resin-bonded or impregnated products such as grinding wheels, impregnated woods, fabrics, felts, glass-fibre and similar products.

### Foodstuffs

Increased use is being made of dielectric heating in many processes connected with foods; these include de-freezing and melting, sterilisation and disinfection, drying of breakfast cereals, dog biscuits, rusks etc., heating of nuts to facilitate shelling and other similar types of application. Although some cooking processes are technically



possible, as for example bread baking, the 'unbrowned' product has so far proved unacceptable to the public and a completely dielectric process uneconomical. When combined with conventional baking, however, as now in the biscuit trade, where dielectric heating is being used to complete the baking of biscuits, it can produce normal biscuits in  $\frac{1}{4}$  to  $\frac{1}{2}$  the usual baking time.

There are in fact so many potential applications of dielectric heating (and these applications are increasing daily as the chemical industry develops new products, as for example synthetic fibres) that the selection given in the present series of data sheets covers only a part of the whole field.

For further information get in touch with your Electricity Board or write direct to the Electrical Development Association, 2 Savoy Hill, London, W.C.2. Telephone: TEMple Bar 9434.

Excellent reference books on electricity and productivity (8/6 each, or 9/- post free) are available—“Induction and Dielectric Heating” is an example.

E.D.A. also have available on free loan in the United Kingdom a series of films on the industrial uses of electricity. Ask for a catalogue.

7148 A



# LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC

INSTRUMENTS AND MATERIALS

SEPTEMBER, 1960

Vol. LXII, No. 371

## Determination of Trace Amounts of Tantalum in Steel

By L. Kidman, C. L. Darwent, and G. White

*English Steel Corporation, Ltd., Sheffield*

*A simple absorptiometric procedure is described for the determination of trace amounts of tantalum in steels. The method can be applied to a wide variety of alloy steels, including the titanium-bearing stainless types, for which existing chemical methods have proved inadequate. Niobium and tantalum are precipitated with phenylarsonic acid from dilute hydrochloric acid solution, zirconium being used as a carrier. The coloured complex between tantalum and pyrogallol is developed in acidified ammonium oxalate solution and its optical density measured by a filter type absorptiometer. The interference from niobium is overcome by control of pH. The method can be extended for the simultaneous determination of both niobium and tantalum in niobium-stabilised steels.*

FOR certain applications in nuclear engineering, the presence of traces of tantalum in steel is highly undesirable because a highly active and long-lived radio-isotope is formed under neutron bombardment. The objection to the formation of this isotope is on two grounds. The first applies to water-cooled reactors, where traces may find their way into the water through corrosion of the primary circuit and, as the primary coolant usually has to circulate outside the main shielding, there is a direct radiation hazard. The second objection is that the steel used in the construction may itself acquire a dangerous level of radioactivity, which means that it will be extremely difficult to arrange access to the components of the reactor for examination or maintenance after the removal of spent elements. For these reasons a limit for tantalum of 0.01% has been set for steels for certain usages, and to date published methods for determining the element have either not been sufficiently sensitive to cope with these extremely low contents, or have not adequately dealt with the interference of metals commonly present in steel.

The paper chromatographic method of Hunt, North and Wells<sup>1,2</sup> proved to have a lower limit of detectability slightly above 0.01%, the mauve-pink colour developed by quinalizarin being masked by the background colour of the pyridine-quinalizarin spray which could not be eliminated by acetic acid. The sample weight for this procedure was limited to 0.2 g., as higher sample weights of steels, when treated with hydrofluoric acid after baking, left a mass of insolubles which did not settle to leave sufficient supernatant liquid from which to extract 0.01 ml. for the chromatographic separation. The sensitivity of the phenyl fluorone method of Luke<sup>3</sup> was found to be no better when a filter type absorptiometer was used to measure the depth of colour developed.

The pyrogallol colour method seemed to offer most possibilities of early success, but neither the technique of Hunt and Wells nor that of Ikenberry, Martin and Boyer<sup>4</sup> satisfactorily solved the problem of titanium interference. The pyrogallol acid complex of titanium

has a colour density some five-and-a-half times greater than that of tantalum. When one considers that the quantities of tantalum being determined are less than 0.01% it is realised that the presence of 0.001% titanium is a serious obstacle, and particularly as this tantalum limit is imposed on the 18/8 type stainless steels containing some 0.75% of titanium.

The determination of titanium on an aliquot from the oxalate extraction of the mixed oxides of niobium and tantalum, and the deduction of the tantalum equivalent from the tantalum figure recorded, is a means used in some laboratories, but this is not satisfactory because of the high conversion factor.

The excellent studies of Ikenberry, Martin and Boyer were accepted as a sound basis for the development of a method which could be readily adopted by steelworks laboratories. This aim was achieved by the complete separation of tantalum from titanium by precipitation from hydrochloric acid solution using phenylarsonic acid. Initially it was found that precipitation of extremely low tantalum contents was not quantitative, but this was overcome by the introduction of zirconium as a carrier.

The effect of temperature on the tantalum-pyrogallol complex was studied, as was the interference of a number of elements known also to form coloured complexes with pyrogallol.

### The Method

#### APPARATUS

All optical densities were measured using the Spekker absorptiometer with mercury vapour lamp, Ilford 601 colour filters and H.503 heat filters. This instrument was chosen, as, being the most widely used in steelworks laboratories, the method could be readily adopted for routine analyses.

#### REAGENTS

**Wash Solution.**—Dissolve 0.5 g. of phenylarsonic acid and 1 g. of ammonium nitrate in hot water and dilute to 1,000 ml.

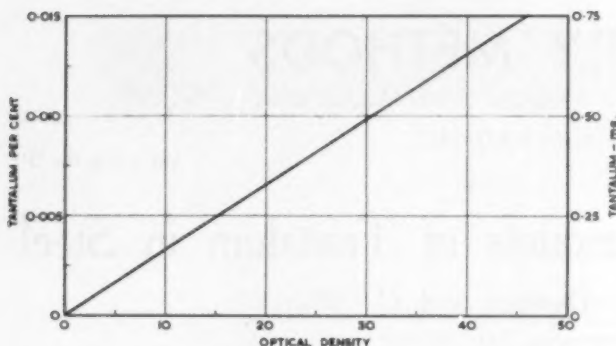


Fig. 1.—Tantalum-pyrogallol complex. 4 cm. cells; 0 → 0.00075g.

**Hydrogen Peroxide.**—100 vols. Analar hydrogen peroxide.

**4% Ammonium Oxalate Solution.**—Dissolve 40 g. of Analar ammonium oxalate in hot water and dilute to 1,000 ml.

**Pyrogallol Solution.**—Dissolve 50 g. of Analar pyrogallol in 100 ml. of cold 2% sulphuric acid (solution takes approximately one hour).

**Phosphoric Acid.**—Dilute 25 ml. of phosphoric acid (S.G. 1.75) to 100 ml.

**Potassium Bisulphate.**—Fused potassium bisulphate.

**Phenylarsonic Acid**

**Zirconium Solution.**—Dissolve 0.3 g. of zirconium nitrate in 50 ml. of 20% hydrochloric acid and dilute to 100 ml.

**Standard Tantalum Solution.**—Fuse 0.1221 g. of tantalum pentoxide (spec. pure) with 5 g. potassium bisulphate. Extract in 4% ammonium oxalate solution. Dilute to 1,000 ml. with ammonium oxalate. (1 ml. = 0.0001 g. Ta; 5, 10, 15 ml. represent 0.005, 0.010, and 0.015% Ta in a 10 g. sample).

#### PROCEDURE

Transfer 10 g. of sample (Note 1) to a 800 ml. squat beaker. Dissolve in 50 ml. hydrochloric acid (S.G. 1.16) and oxidise by dropwise additions of nitric acid.

Evaporate to dryness (Note 2) and bake lightly. Redissolve the cake in 40 ml. of hydrochloric acid (S.G. 1.16). Dilute to 100 ml. with water and filter through a paper pulp pad, washing with hot 5% hydrochloric acid. Ignite the pad in a platinum dish, treat with 8% oxalic acid and hydrofluoric acid. Dry and ignite at 800° C. Fuse the residue with 2 g. potassium bisulphate, extract the melt with 5 ml. of 50% sulphuric acid and add to the filtrate.

Add 5 ml. of zirconium solution, 10 ml. of hydrogen peroxide (100 vols.) and dilute to 400 ml. with hot water. Bring to the boil and boil for 3 minutes. Add phenylarsonic acid as a hot solution (1 g. dissolved in 20 ml. of hot water), stir well and add a small piece of paper pulp. Set aside for one hour at 90° C.

Filter through a tight pulp pad, washing at least six times with hot wash solution. Transfer the precipitate and pad to the beaker and warm with 30 ml. of hydrochloric acid to redissolve. Dilute to 300 ml., add 5 ml. of hydrogen peroxide and boil for 3 minutes. Reprecipitate

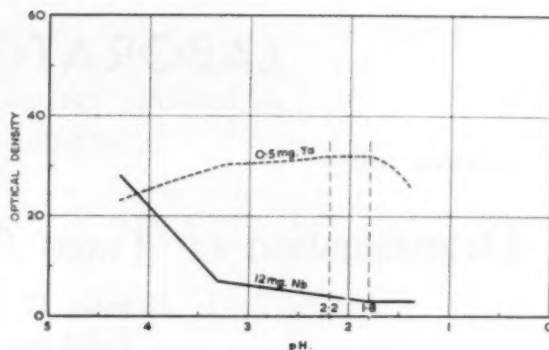


Fig. 2.—Effect of pH. Tantalum- and niobium-pyrogallol complexes.

using 0.5 g. of phenylarsonic acid dissolved in hot water. Stand for one hour at 90° C.

Filter through a tight pulp pad and wash six times with wash solution. Ignite the precipitate in a porcelain dish at a temperature exceeding 800° C. After ignition transfer the precipitate to a platinum dish (Note 3) and fuse in 5 g. of potassium bisulphate at low temperature. Extract the melt in 4% ammonium oxalate solution, warming to dissolve. Cool and dilute to 100 ml. with 4% ammonium oxalate solution.

Transfer a 50 ml. aliquot to a 125 ml. conical beaker. Add 4 ml. of 25% phosphoric acid and 20 ml. of ammonium oxalate solution. Adjust the pH of the solution to 2.1 by either phosphoric acid or ammonia using a sensitive pH meter. Add 20 ml. of pyrogallol solution and dilute to 100 ml. with ammonium oxalate solution. Stand in a water bath at 26° C. (Note 4) for 15 minutes for full colour development. Measure the absorption immediately in 4 cm. cells using an absorptiometer with mercury vapour lamp and Ilford 601 filters (Note 5). (For a spectrophotometer the optimum wavelength is 430 mμ.)

#### CALIBRATION

Into 150 ml. conical flasks transfer varying aliquots from the standard tantalum solution to give tantalum contents of up to 1.5 mg. To each aliquot add 5 g. potassium bisulphate and approx. 70 ml. of 4% ammonium oxalate solution. Warm until clear. Cool and dilute to 100 ml. using a graduated flask. Pipette a 50 ml. aliquot for colour development. To each aliquot add 4 ml. of 25% phosphoric acid and 20 ml. ammonium oxalate solution. Adjust the pH of the solution to 2.1 with either phosphoric acid or ammonia, using a sensitive pH meter, and add 20 ml. of pyrogallol solution. Using a graduated flask, dilute the solution to 100 ml. with 4% ammonium oxalate solution. (The pH of this solution lies between 1.8-2.0 if previously adjusted accurately.)

Develop the colour in a water bath at 26° C. for 15 minutes. Measure the absorption immediately in 4 cm. cells using an absorptiometer with mercury vapour lamp and Ilford 601 filters. A good linear relationship is obtained (Fig. 1).

#### NOTES

- (1) The method was developed for tantalum contents up to 0.015%. The range may be considerably

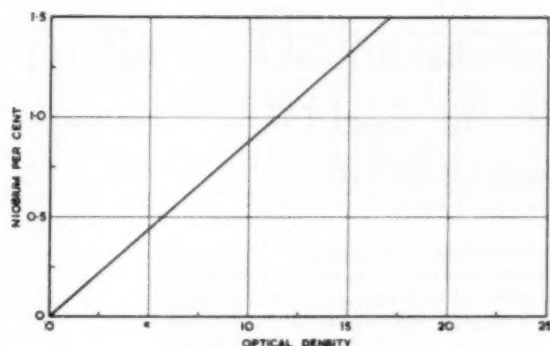


Fig. 3.—Niobium-pyrogallol complex. 4 cm. cells; pH 2; 1% Nb  $\equiv$  0.004% Ta.

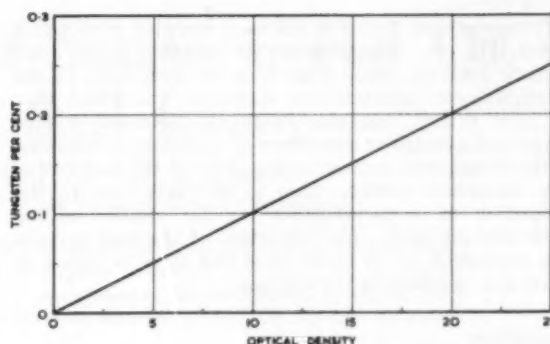


Fig. 4.—Tungsten-pyrogallol complex. 4 cm. cells; pH 2; 0.05% W  $\equiv$  0.00125% Ta.

extended (to cover tantalum contents up to 0.1%) by reducing the sample weight and cell size.

- (2) For low silicon steels containing only residual amounts of titanium and no insoluble carbides, omit the second paragraph under "Procedure." Add 30 ml. of hydrochloric acid to adjust the acid strength to 10%. Add 5 ml. of zirconium solution ... (Continue as in "Procedure.")
- (3) If platinum dishes are used for the ignition, they can be seriously attacked, particularly at low temperatures, by the arsenical complex.
- (4) Any temperature between 20° C. and 30° C., at which the solution can be accurately controlled, is suitable. 26° C. was found to be most convenient using the thermostatically controlled bath available in these laboratories. Using higher temperatures it was found that the rate of fall of temperature was too rapid for accurate measurement of colour density.
- (5) Use the zero setting method (BISRA Special Report No. 55, P. 4, Method 16) with the reagent blank as compensating solution.

### Experimental

#### STABILITY OF THE COMPLEX

0.25 mg. and 0.50 mg. of tantalum and a reagent blank were coloured by the above procedure. Using the blank as a compensating solution the optical density was read at fifteen-minute intervals up to one hour. Providing the temperature of the measured solution was within the range of 26° C.  $\pm$  1°, the optical density remained constant.

Amount of Tantalum	15 min.	30 min.	45 min.	60 min.
0.25 mg.	15	15	15	15
0.50 mg.	30 <sup>a</sup>	30 <sup>a</sup>	30 <sup>a</sup>	30 <sup>a</sup>

Ikenberry, Martin and Boyer state that the optical density of the complex increases with decrease in temperature. This was proved to be true when the absorbencies of these solutions were measured at lower temperatures.

Amount of Tantalum	26° C.	23° C.	21° C.
0.25 mg.	15	16	17
0.50 mg.	30 <sup>a</sup>	32 <sup>a</sup>	34

The solutions were held at 23° C. for one hour and the absorbencies recorded above remained constant through-

out. It can be assumed therefore, that, provided the optical density is measured at the temperature of colour development, the complex is stable.

#### EFFECT OF VARIOUS ELEMENTS ON THE COLOURED COMPLEX

##### Niobium

Ikenberry, Martin and Boyer report that at pH 1.8–2.2 the optical density of the pyrogallol-tantalum colour is at a maximum whilst that between niobium and pyrogallol is at a minimum. This statement was checked using the absorptiometer, and the results recorded graphically (Fig. 2) proved it to be true. The effect of niobium was then tested using the final procedure.

Additions of niobium equivalent to 1.5% (0.15 g.), were made to the final 100 ml. of solution prior to colour development. From the graph (Fig. 3) it is seen that niobium gives a slight positive absorption, 1% niobium being equivalent to approximately 0.0037% tantalum. As the graph is linear it can be used as a simple correction graph. However, for trace tantalum determinations, e.g. in steels not stabilised by additions of ferro-niobium, niobium interference can be ignored.

##### Zirconium

From solutions of pure zirconium metal in *aqua regia*, various aliquots representing up to 1% (0.1 g. zirconium) were added to the final 100 ml. solution prior to colour development. As no interference was experienced, zirconium was selected as a carrier for the lower tantalum contents.

##### Molybdenum

Molybdenum forms a coloured complex with pyrogallol under the conditions used for the formation of the tantalum complex. As little as 0.01% molybdenum gives a tantalum equivalent of 0.060%. To test the separation of molybdenum from tantalum by the phenylarsonic precipitation, amounts of molybdenum equivalent to 2% were added to solutions containing various percentages of tantalum. The recommended double precipitation procedure was carried through and the tantalum complex developed under the conditions stated. Tantalum recovery was quantitative, the phenylarsonic acid giving complete separation of tantalum from molybdenum contents up to 2%.

## Tungsten

Tungsten also forms a coloured complex with pyrogallol (Fig. 4). The presence of tungsten in the final oxalate solution would arise from its hydrolysis at the phenylarsonic precipitation stage. It was found that at least 0.25% tungsten could be tolerated at this stage and a complete separation of tantalum be obtained without tungsten contamination, even in the presence of the zirconium carrier. This is probably due to the formation of a pertungstate on the addition of the hydrogen peroxide. The formation of this salt permits the separation to be made, provided there is excess of hydrogen peroxide in the solution.

## Vanadium

Vanadium is another element which forms a colour with pyrogallol, 0.01% vanadium giving a tantalum equivalent of 0.04%. However, as with molybdenum, the double precipitation procedure proved to eradicate the interference of vanadium when present in amounts up to 2%.

## Titanium

The solution prepared for the development of the tantalum-pyrogallol colour must be entirely free from titanium, as this element seriously interferes, forming a coloured complex having an optical density some five-and-a-half times greater than that of tantalum. Oxides of niobium and tantalum recovered from steel solutions by normal hydrolysis procedures contain small amounts of titanium even after purification.

Phenylarsonic acid has been known for some time as a more or less specific reagent for zirconium, even in the presence of titanium. Any tantalum present in the solution will be precipitated with the zirconium. To test the precipitation of tantalum and also its separation from titanium using this reagent, the following tests were carried out.

Varying amounts of tantalum over the range 0.1-5 mg. were added to solutions of 0.1 g. of titanium in 40 ml. of hydrochloric acid. After diluting the solutions to 400 ml., 10 ml. of hydrogen peroxide were added to oxidise the titanium and the solutions boiled for 3 minutes. The reagent was added as hot aqueous solutions of 1 g. of phenylarsonic acid, and the solutions allowed to stand for one hour at a temperature of approximately 90° C. to complete precipitation. After filtration the precipitates were washed and transferred to the original beakers to be redissolved in 30 ml. of hydrochloric acid. Reprecipitation under similar conditions was carried through, but using only 0.5 g. of reagent. After filtration the precipitates were ignited at a temperature exceeding 800° C., fused with 5 g. of potassium bisulphate, extracted in ammonium oxalate solution, and diluted to 100 ml. with the same solution. From this solution a 50 ml. aliquot was taken for colour development, the remaining 50 ml. being tested for titanium.

It was found that although titanium had been successfully removed the precipitation of tantalum was incomplete, the recovery of low contents being only about 50%. This was possibly due to the small amount of tantalum involved in the precipitation and further tests were made in which 3 mg. of zirconium were used as a carrier, the zirconium being added in the form of zirconium nitrate solution.

TABLE I.—APPROXIMATE PERCENTAGE COMPOSITION OF THE TEST SAMPLES

B.C.S. No.	C	Si	Mn	Ni	Cr	Mo	V	W	Nb+Ta	Ti
219/2	0.31	0.30	0.64	2.5	0.8	0.43	0.02	—	—	—
235/1	0.04	0.60	0.80	8.2	18.4	0.04	0.04	—	—	0.26
246	0.06	—	—	12.0	18.8	2.9	—	0.22	0.82	—
252	—	0.25	0.02	4.1	0.2	0.01	0.46	—	—	—
261	0.08	0.39	0.66	13.1	17.2	0.02	0.04	0.71	—	—
274	0.18	0.32	0.31	0.13	0.19	0.07	0.02	0.04	—	0.10

Tantalum Added	Tantalum Recovered
0.50 mg.	0.50 mg.
0.75 mg.	0.75 mg.
1.00 mg.	1.00 mg.
1.50 mg.	1.50 mg.

From these figures it can be seen that the recovery of tantalum was quantitative and the separation from titanium was complete.

## RECOVERY OF TANTALUM IN THE FINAL PROCEDURE

To 10 g. samples of pure iron varying amounts of tantalum were added to give contents ranging from 0 to 0.015%. Titanium solution was then added to each of these to give a 1% titanium concentration. After dissolution in hydrochloric acid the samples were carried through the recommended procedure. Tantalum recoveries were measured using the blank as a compensating solution.

Tantalum Added	Tantalum Recovered
0.005% (0.50 mg.)	0.005%
0.0075% (0.75 mg.)	0.0072%
0.010% (1.00 mg.)	0.0095%
0.015% (1.50 mg.)	0.0145%

As there are no known British Standard steels for trace tantalum, a synthetic steel solution was prepared to check the recovery of tantalum under what were considered to be the worst conditions. This solution contained 0.2 g. of molybdenum, 0.2 g. of vanadium, 0.1 g. of titanium, 0.25 g. of tungsten, the balance of 10 g. being made up with pure iron.

Tantalum Added	Tantalum Recovered
0.005% (0.50 mg.)	0.004% (0.40 mg.)
0.0075% (0.75 mg.)	0.008% (0.80 mg.)
0.010% (1.00 mg.)	0.010% (1.00 mg.)
0.015% (1.50 mg.)	0.0145% (1.45 mg.)

## ANALYSIS OF STEELS

Using the final procedure the tantalum contents of a number of British Chemical Standards were determined, with the following results.

B.C.S. No.	Tantalum %
219/2	0.0015
235/1	0.001
246	0.028
252	0.001
261	0.043
274	0.008

The compositions of these Standards are given in Table I.

(Continued on page 130)



# Rapid Determination of Sulphur in Fuel Oils Used in the Steel Industry

By W. B. Boyes and H. C. Wilkinson

*British Coke Research Association, Coke Research Centre, Chesterfield.*

*A study has been made of the application of the calorimeter-bomb method and the Sheffield high-temperature method to the determination of the sulphur content of fuel oils. In both cases problems arose with regard to the efficiency of absorption of the combustion products. Both modified procedures provide a rapid and reasonably accurate method for the determination.*

THE sulphur content of motor fuels and of kerosine for domestic use is kept low so as to minimise the tendencies towards corrosion and offensiveness on combustion. On the other hand, the sulphur content of fuel oils has not in the past been regarded as being of great significance: the value for heavy fuel oil may be 2% or more. Today, however, the increased use of fuel oil in open-hearth furnaces makes the sulphur content of the oil a matter of importance, for it is known that the amount of sulphur absorbed by the steel is proportional to the sulphur content of the fuel. For steel for certain uses (e.g. making into sheet, where the tendency to crack increases with the sulphur content of the steel) it is therefore desirable to have fuels of low sulphur content. Considerations of atmospheric pollution also make it desirable to limit the sulphur content of the fuel oils which are used in large quantities for open-hearth furnaces and power stations.

For determining the sulphur in light fuel oils some modification of the lamp method is usually used, the fuel being burnt in a "lamp" and the oxides of sulphur in the waste gases absorbed in hydrogen peroxide and determined titrimetrically. The application of this technique to heavy oils would present certain difficulties. In this paper a report is given of work carried out on the application of two other methods: the calorimeter-bomb method<sup>1</sup> and the Sheffield high-temperature method,<sup>2,3</sup> both of which have been standardised for the determination of sulphur in solid fuels and in both of which the combustion process takes only 10 min. In both cases problems arose with regard to the efficiency of absorption of the combustion products.

## Calorimeter-Bomb Method

It has been shown<sup>4</sup> in the laboratories of the British Coke Research Association that complete combustion of liquid fuel in a calorimeter bomb in a standard time may be ensured by fixing the dimensions of the crucible in which the fuel is burnt according to the volatility and using a suitable pressure of oxygen. For gas oil a crucible of top diameter, bottom diameter, and height all  $\frac{3}{8}$  in. and an oxygen pressure of 35 atm. (gauge) were recommended. The crucible used was made of nickel-chromium but platinum would also be suitable.

One of the disadvantages of the calorimeter-bomb method is that, in addition to all the sulphur forming sulphuric acid, some of the nitrogen in the system forms nitric acid, the amount being a function of the heat release and the bomb capacity. It is possible to determine a nitric acid "blank" by using the same heat release from benzoic acid (which is free from sulphur)

burnt under the conditions used for the fuel; the blank is fairly high. In the ordinary calorimetry of solid fuels the original atmosphere of air in the bomb is retained so that the oxides formed from the nitrogen will ensure complete conversion of sulphur to sulphur trioxide, for the validity of the sulphur correction is dependent on the assumption that this occurs. When the sulphur is to be determined from the bomb washings by the gravimetric method the same requirement must be fulfilled. If a titrimetric finish is to be used, however, it is unimportant whether the sulphur is present as sulphurous or sulphuric acid, so that it seemed that it would be permissible to reduce the blank by displacing the original atmosphere of air. An easy way of doing this was to fill the bomb with oxygen at 30 atm. (gauge) pressure and then to release that pressure; the amount of nitrogen in the bomb was thus reduced to 1/31 of the original quantity. The bomb was then refilled with oxygen to the required pressure. Small and consistent blanks were obtained on burning benzoic acid under these conditions.

The calorimeter bomb used was a Griffin-B.C.R.A. bomb, of  $\frac{1}{2}$  litre capacity and with a quick-release (Schrader) valve, modified for controlled release by means of an adaptor with a needle valve screwed on to the oxygen-filling boss (as in Reference 5, Fig. 1b). The usual 1 ml. of water was placed in the bomb before closing and filling with oxygen. For the sulphur left in the gaseous phase, two absorbers containing hydrogen peroxide were used. At first a No. 2 sintered-glass bubbler was used in the first absorber with a plain tube in the second, but it was found to be preferable to use a No. 2 sintered-glass bubbler in each to enable a rate of gas flow of about 1 litre/min. to be achieved. Since the total volume (with an oxygen pressure of 35 atm., gauge) was about 12 litres, the absorption took 12 min. When the flow of gas had ceased, the contents of the absorbers were washed into a conical beaker, the bomb washings added, and the liquid boiled to remove carbon dioxide so that the acidity could be determined by titration with sodium borate using methyl red/methylene blue as indicator. The chlorine content of the oils used was usually negligible, but for a cutting oil a chlorine correction had to be made. The first titration gave total acidity; on adding 20 ml. of saturated mercuric cyanide solution (usually a sufficient excess) the sodium chloride was converted stoichiometrically to sodium hydroxide, which was then determined by titration with sulphuric acid. The nitric acid blank was deducted from the determined total acidity (corrected, when necessary, for hydrochloric acid) to obtain the amount of sulphuric acid, whence the sulphur content of the fuel

TABLE I.—SULPHUR CONTENT (%) OF FUEL OILS BY BOMB AND HIGH TEMPERATURE METHODS

Fuel	Sulphur Content (%)			A—C (or B)
	Bomb Method A	High-Temperature Method <sup>a</sup>		
		B	C	
Hard Pitch .. ..	0.73	0.71	—	0.01†
Fluxed Pitch .. ..	1.04	0.91	0.96	0.08
Pitch Creosote O ..	1.03	0.97	1.03	0.00
Pitch Creosote S ..	0.94	0.78	0.78	0.16
Heavy Fuel Oil ..	1.19	1.27	—	—0.08
Lubricating Oil ..	0.16	0.16	—	0.00
Derv .. ..	0.35	0.39	0.38	—0.03‡
Creosote Oil .. ..	1.05	0.97	1.03	0.02

<sup>a</sup> B = one absorber; C = two absorbers.

† Rate of flow 300 ml./min. }

‡ Rate of flow 900 ml./min. }

could be calculated. The time for the complete determination was about half an hour.

### High-Temperature Method

In the Sheffield high-temperature method for coal and coke, 0.5 g. of fuel, covered with alumina, is burnt in oxygen supplied at a rate of 300 ml./min. The maximum temperature in the furnace is 1,350° C., this and the alumina cover ensuring that all sulphates are decomposed, an important consideration for coals with basic ashes. The ash of liquid fuels is usually negligible in amount and fixation of sulphur is unlikely, so that the temperature of 1,350° C. may be unnecessary for fuel oils. However, this standard temperature was retained because the method is now being extensively adopted in the steel industry for the determination of sulphur in coke, and it was considered desirable to modify the standard procedure as little as possible.

For ordinary coals and cokes a standard pushing schedule ensures that the boat containing the fuel is systematically pushed into the centre of the hot zone over a period of 6 min.; the pushes are given at intervals of 1 min. and each takes the boat through a distance of 1½ in. For more volatile materials it is necessary to reduce the rate of pushing in order to avoid errors due to the more rapid evolution of volatile matter. A similar modification had to be adopted for fuel oils. The boat was inserted into the tube and left at the end for 2 min.; after each of the next five intervals of 1 min. it was pushed ½ in.; after a further period of 1 min. it was pushed to the centre of the hot zone and left for 2 min.; thus the total time in the furnace was 10 min.

The boats used for solid fuels are made of unglazed porcelain and are 2½ in. long, ½ in. wide and ¾ in. deep. For the fuel oils boats of both glazed and unglazed porcelain were used; both were 4½ in. long and the widths were ½ and ¾ in., respectively. Both were satisfactory. Approximately 0.5 g. of fuel was added dropwise to a layer of alumina spread in the boat, which was then re-weighed to find the exact weight of fuel; the fuel was then covered with 2½ g. of alumina.

For hard pitch it was found that the rate of flow of oxygen used for solid fuels was adequate, but for all the other materials tested the rate had to be increased to

TABLE II.—SULPHUR CONTENT (%) OF FUEL OILS BY HIGH-TEMPERATURE METHOD

Fuel	B.P. Value (A)	High-Temperature Method (B)	A—B
Fuel Oil .. ..	1.95	1.93	0.02
Lubricating Oil .. ..	0.61	0.56	0.05
Cutting Oil .. ..	2.24	2.29*	—0.05
Marine Diesel Oil .. ..	1.15	0.99	0.16
Gas Oil .. ..	1.21	1.09	0.12

\* An abnormally high chlorine content necessitated the use of at least 40 ml. of mercuric oxycyanide solution.

600 ml./min. and sometimes, to prevent incomplete combustion, to 900 ml./min. When the rate of flow of oxygen was too low black particles of carbon appeared in the absorbers. For a rate of flow of 300 ml./min. a single absorber with a No. 3 porosity sintered-glass bubbler gives complete absorption. For the faster flow used for fuel oils two absorbers, each with a No. 3 porosity sintered-glass bubbler, were tried when the result obtained using one absorber was low in comparison to that obtained by the calorimeter-bomb method. The results are recorded in Table I.

It appeared that two absorbers were desirable where there was a fairly volatile fraction present; with such a precaution the agreement between the two methods was reasonably satisfactory except for pitch creosote S. However, as neither could be accepted as necessarily giving accurate results, a series of tests was done on a number of oils supplied, together with data on the sulphur contents, by the British Petroleum Co., Ltd. The results are recorded in Table II.

These data suggested that the high-temperature method gave results which were slightly too low. Taking into account the data of Table I, it seemed possible that the bomb method was slightly more accurate.

### Conclusions

The calorimeter-bomb method with a titrimetric finish is a rapid method for the determination of sulphur in fuel oils. The Sheffield high-temperature method, which is used at many steel works for the determination of sulphur in coke, can be used for the determination of the approximate sulphur content of fuel oils; the approximation may be adequate for most purposes.

### Acknowledgments

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## Determination of Tantalum

(Continued from page 128)

### Conclusions

A method has been developed in which the precipitation of tantalum by phenylarsonic acid from a 10% hydrochloric acid solution and the formation of a coloured complex with pyrogallol can be applied to the determination of trace amounts (<0.015%) of tantalum in steel. By reduction of the initial sample weight the range can be extended to cover niobium-stabilised steels. In this type of steel, niobium and tantalum can be determined on the same sample, the niobium determination depending on the niobium pyrogallol colour reaction in alkaline solution.<sup>1</sup>

### Acknowledgments

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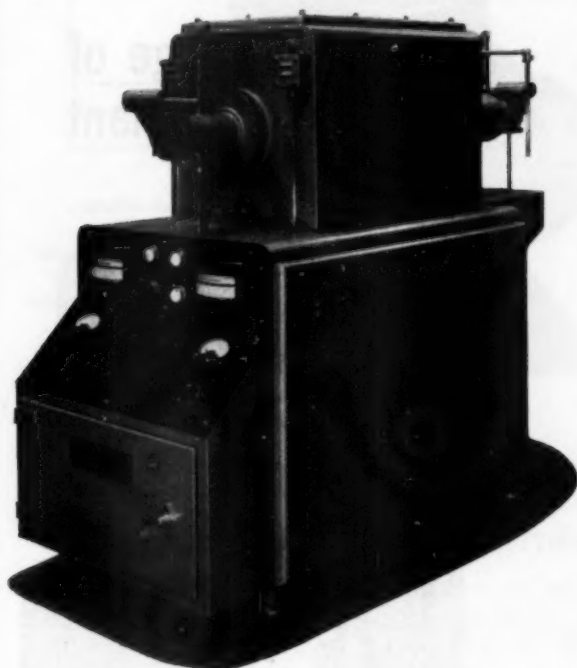
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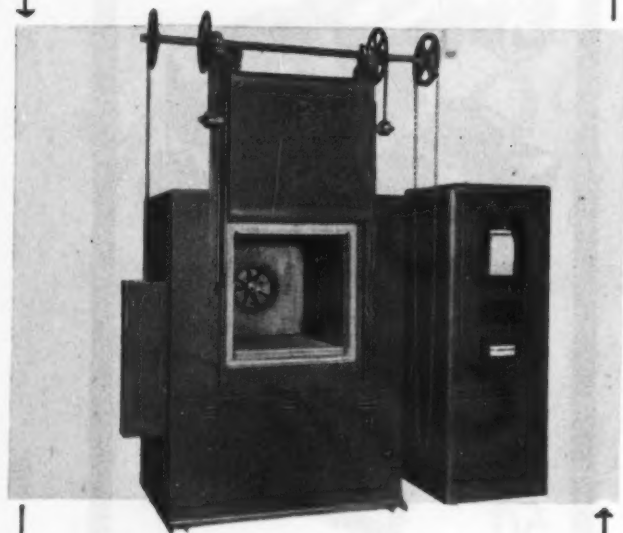
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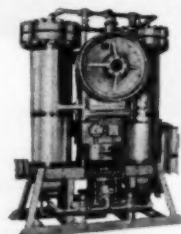
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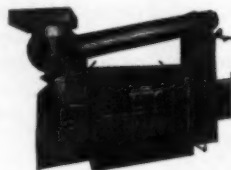
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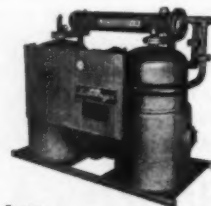
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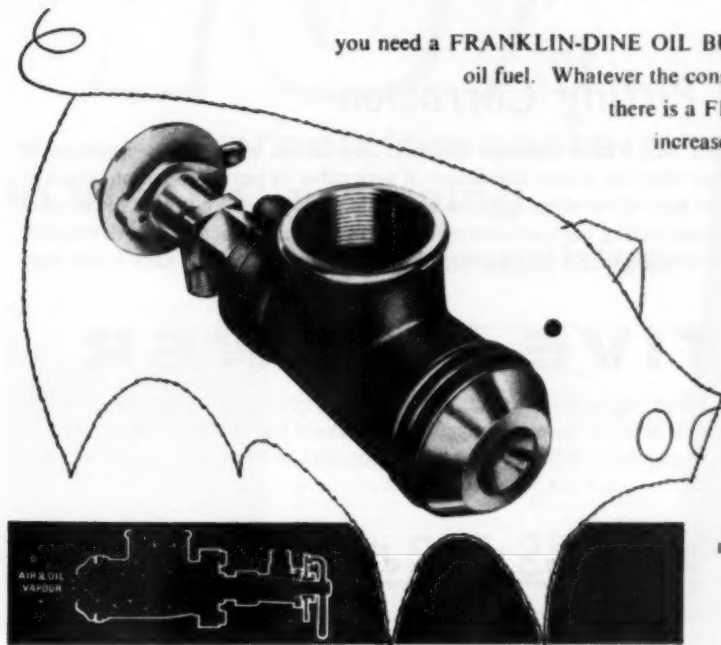
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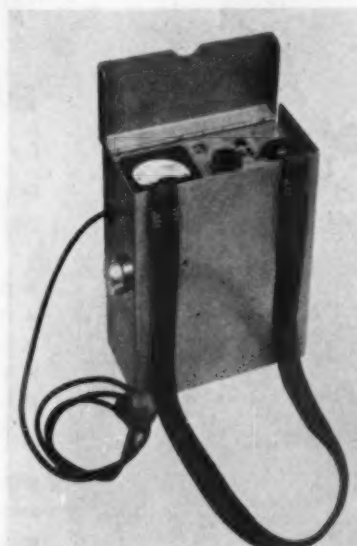
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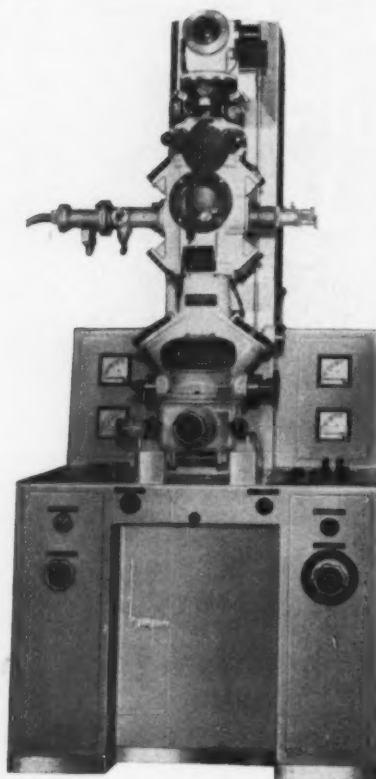
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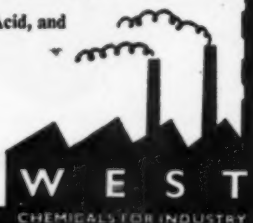
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## BICC

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required for the Metallurgical Laboratory of the Wire Mill Division at Prescott Works.

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Applications to: **Works Personnel Officer,  
Ref. No. P.455,  
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**Firth Brown Tools, Ltd.  
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Written applications should be addressed to the Labour Manager, Firth Brown Tools, Ltd., Speedicut Works, Carlisle Street East, Sheffield, 4.

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Salary will be appropriate to the qualifications and experience of the successful candidates. Pension and assurance schemes are in operation and, in appropriate cases, assistance can be given for housing. Applications, which will be treated in confidence, should give details of age, qualifications and experience. They should be addressed to the General Manager, Development and Research Department, The Mond Nickel Co., Ltd., Thames House, Millbank, London, S.W.1. Please mark envelope "Confidential, L.86/87."

## GKN

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